INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

A STUDY OF FACTORS PERTINENT TO HARDWOOD PULP UTILIZATION PHASE I: FIBER PROPERTIES, SHEET STRUCTURE, AND SHEET PROPERTIES

Project 2502

Report Two

A Progress Report

to

MEMBERS OF GROUP PROJECT 2502

February 23, 1968

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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A STUDY OF FACTORS PERTINENT TO HARDWOOD PULP UTILIZATION
PHASE II. FIBER PROPERTIES, SHEET STRUCTURE, AND SHEET PROPERTIES

SUMMARY

This project has been concerned with three aspects of importance in better utilization of hardwood pulps. Phase I dealt with factors affecting fiber strength. In order to utilize fiber strength most effectively to produce good sheet properties a better understanding of the relationship of fiber properties and sheet structure to sheet properties is needed, with tearing resistance being one of the more important sheet properties in the use of hardwood pulps. During the preceding project an equation was developed to relate tearing resistance of a sheet to fiber strength, fiber length distribution, and sheet structure. Phase II of the current project was directed at the development of more quantitative relationships between fiber properties, sheet structure, and sheet properties using this tear equation as a starting point. The program was planned primarily to test the tear equation but to provide data also for more general considerations.

In the current work, the application of this equation has not been successful, possibly because such an application is premature, until we have a better understanding of the tearing process. It was possible to obtain a good fit of the data to the equation but the equation did not seem able to predict fiber rupture frequencies and did not seem to lend itself to the development of a better understanding of pulp properties.

One observation of interest was the apparent relationship between extensional stiffness and specific surface. Extrapolation of the curves to zero extensional stiffness resulted in intercept values approximately equal to the

specific surface area determined experimentally for completely unbonded fibers. Data for the three pulp species (white oak, aspen, and western softwood) gave straight lines of equal slope on a semilogarithmic plot, suggesting that extensional stiffness is dependent upon relative bonded area and independent of pulp species. This observation may be specific to the experimental conditions used, especially the use of pulps from which fines have been eliminated. If it can be confirmed by further work and applied on a more general basis it could provide a useful measure of relative bonded area.

INTRODUCTION

Progress Report One discussed briefly the origin of the current project as an outgrowth of Group Project 2070, "Strength Properties of Hardwood Pulps" and some of the pertinent factors. A proposal for continued research was submitted in May, 1964 with an emphasis on three factors important to better hardwood pulp utilization. That proposal was approved by 21 companies and a project initiated August 1, 1964. In 1967 a majority of the sponsors approved a request for extension of the contract period from August 1 to December 31, 1967 to permit carrying the work to a more logical stopping point. Progress Report One covered the work done during the initial contract period on Phase I, "Factors Affecting Fiber Strength." This report covers the implementation during the original contract period of the approach outlined in the proposal for Phase II on "Fiber Properties, Sheet Structure, and Sheet Properties."

Project 2070 studied a number of aspects of the strength properties of hardwood pulps. Phase I of this project was aimed at providing information on the factors controlling fiber strength. In order to utilize fiber strength most effectively in developing good sheet properties, more information on the relationship of fiber properties and sheet structure to sheet properties is required. Tearing resistance is one of the critical properties in the use of hardwood pulps and therefore is one sheet property of primary interest. During Group Project 2070, an equation has been developed by which tearing resistance was analyzed in terms of fiber strength, fiber length distribution, and sheet structure. Experimental application of the equation at the conclusion of Project 2070 was encouraging from the standpoint that values predicted for frequencies of fiber rupture and of pull-out were in reasonable agreement with data found in the literature.

Further evaluation of this equation was suggested as a starting point for the development of more quantitative relationships between fiber properties, sheet structure, and sheet properties, including actual measurement of frequencies of fiber rupture and pull-out for comparison with calculated values. Some of the techniques required, such as those for fiber strength and fiber length distribution, are fairly well recognized. Other techniques required further evaluation and are discussed in this report. The program was planned primarily to test the tear equation but at the same time to provide data relevant to a more general analysis of Elmendorf tear, in-plane tear, and tensile strength. Two hardwood and one softwood pulps were beaten to three different levels, classified to remove fines, and formed into handsheets. Various levels of sheet structure were obtained by varying the wet pressing of each of these sets of handsheets. The resulting sheets were then tested for appropriate properties and compared with fiber length, fiber strength, and characterizations of sheet structure.

EXPERIMENTAL PROCEDURES AND RESULTS

PULPS AND HANDSHEETS

Two hardwoods, white oak and aspen, were cooked to low lignin contents by a kraft process and bleached with a chlorine-caustic extraction-chlorine dioxide treatment. The resulting pulps, along with a commercial western softwood bleached sulfite pulp, were refined in a Valley beater to 3 different levels and classified on the IPC web former to remove fines. Classified samples were dewatered to consistencies of 30-35% and stored in polyethylene bags at 40°F. until used. Details on cooking and bleaching of the hardwood pulps and on the material balances for the classifications are given in Appendix I.

All handsheets were prepared according to TAPPI Method T 205 with the exception of the wet pressing conditions which were varied in order to attain different levels of bonded area. Based upon previous work at the Institute the following conditions were selected:

WET PRESSING CONDITIONS

	Pressing Load, p.s.i.	Wet Pressing Conditions
Set 1	0	Couch with lightweight Lucite roll; do not use a couch plate
Set 2	1	Couch with a hollow brass roll; do not use a couch plate. Place cover and weight (31 lb. total) on sheets for 5 minutes.
Set 3	5	Couch with a hollow brass roll; press at 5 p.s.i. for 5 minutes
Set 4	10	Couch with a hollow brass roll; do not use a couch plate. Press at 10 p.s.i. for 5 minutes.
Set 5	25	Couch with a couch plate and a solid brass roll. Press at 25 p.s.i. for 5 minutes.
Set 6	50	Standard TAPPI couching and pressing.

Following wet pressing the sheets were dried on rings in the standard manner.

CHARACTERIZATION OF FIBER PROPERTIES

A measure of fiber strength was obtained by use of the zero-span tensile test as described by Wink and Van Eperen (1) and the results are expressed as breaking length in meters in Appendix I, Table VI. Changes in zero-span strength have been found in the past as a function of beating and of wet pressing. To the extent that better bonding (either through beating or increased wet pressing) decreases slippage of the fibers in the zero-span jaws, the higher values may be considered to be more nearly a true indication of fiber strength. However, it is also possible that increased bonding may result in increased shrinkage on drying and, since these sheets were dried under restraint, in drying under increased stress. It has been shown, particularly by Jentzen (2), that fibers dried under stress possess greater tensile strength than fibers dried with no restraint. It is possible that the low values at very low wet pressing are incorrect because of poor gripping of the fibers or because of damage to the very tender wet sheets made without adequate wet pressing.

Fiber length distributions were run by the projection method. Distributions are shown in Appendix I, Fig. 10 to 15 and the arithmetic average fiber lengths of each pulp are shown in Appendix I, Table VII.

Coarseness measurements were made by a scan line technique on a very thin sheet [comparable to the 2-D sheet of Kallmes and Corte (3)]. This method is similar in concept to TAPPI Method T 234 but utilizes somewhat different techniques. Very thin sheets were formed using 0.05 g. of ovendry pulp per sheet in a TAPPI standard sheet mold on a 200-mesh nylon screen. Random areas of the sheet were viewed through a 20X microscope, and fiber intersections with a scan line of known length superimposed by an eyepiece were counted. From this the fiber coarseness in mg. per 100 meters could be calculated by the relationship:

$$C = \left(\frac{2}{\pi}\right)\left(\frac{W}{A}\right)\left(\frac{L}{N}\right)$$

where: $\underline{W}$ = the weight of the sheet

$\underline{A}$ = the area of the sheet

$\underline{L}$ = the length of the scan line

$\underline{N}$ = fibers crossing the scan line

Coarseness values for the three experimental pulps are shown in Appendix I, Table VII. The coarseness of the whole unbeaten hardwoods seemed to be high in relation to the classified fibers. A plausible explanation for this is that a substantial portion of the fines present in the whole pulp were either lost in the sheet-forming process or overlooked in the scan line observations.

MEASUREMENT OF SHEET STRENGTH

In general, the strength characteristics of the handsheets were measured by conventional techniques with a few exceptions and the detailed results are given in Appendix II, Table VIII. Tensile tests were conducted on the Instron tester using 1-inch wide specimens with an initial span of 4 inches and a cross-head speed of 1 inch per minute. Breaking strength, stretch at break, and extensional stiffness were determined. Extensional stiffness is determined as the slope of the initial portion of the tensile load-deformation curve. At constant basis weight, it is directly proportional to tensile elastic modulus expressed on a solid cross-sectional area basis.

Elmendorf tearing strengths were measured by the usual 5-ply test as specified in TAPPI Method T 205 and also by a single-ply test using a tester of greater sensitivity. The effect of multiple plies has been discussed by Wink (4). In this work the 5-ply test was found to give somewhat higher values and in many of the tests the outside plies tended to delaminate.

Tearing resistance was also evaluated by the in-plane tear in which the forces promoting the fracture of the sheet are applied in the plane of the sheet as opposed to the Elmendorf test where the forces are perpendicular to the initial plane of the sheet. The development of this test was described by Van den Akker, Wink, and Van Eperen (5).

CHARACTERIZATION OF SHEET STRUCTURE

After considering several possibilities, relative bonded area was chosen as the principal characterization of sheet structure. One approach to the estimation of relative bonded area is the measurement of unbonded area of a sheet by the dynamic gas adsorption technique (6). The unbonded area of a specimen is calculated from the nitrogen adsorption from a flowing gas mixture at liquid nitrogen temperatures and the desorption when the specimen temperature is raised to 21°C. (the adsorption and desorption values should agree). In order to determine relative bonded area from these measurements a measurement of completely unbonded area also must be obtained.

Methods employed in obtaining water-dried unbonded fiber samples have been subject to criticism because they often tend to produce damaged, classified, or bonded samples. A measure of success has been attained by using very dilute suspensions of fibers (1 g. per 100 liters) on the I.P.C. web former and carefully doctoring the dried fibers from the first drying cylinder kept barely warm. This technique was quite suitable for this work for although the fibers became classified in passing over the fourdrinier wire, the classification occurred in the same manner used to prepare the classified fibers for handsheet production. Specific surface area data for handsheets and unbonded fiber are given in Appendix II, Table IX.

When tensile breaking lengths are plotted as a function of specific surface, extrapolation to zero tensile strength indicates unbonded areas in agreement with the unbonded fiber area measurements. (See Appendix II, Fig. 16 to 18.) From this it appears that the unbonded fiber areas are of reasonable magnitude and that the technique of obtaining unbonded fibers provided representative samplings. Relative bonded area calculations based upon these data should be acceptable estimates knowing that the surface area measurements may include internal areas of the fibers accessible to the nitrogen so that relative bonded area estimates include inter- and intrafiber bonding.

MEASUREMENT OF FIBER RUPTURE AND PULL-OUT FREQUENCIES

In this work, the determination of fiber rupture versus pull-out frequencies is made by observing, at a reasonable magnification, fibers tagged with dye in the zone of fracture. One determines the proportion of fibers that pull out of one side or the other and the proportion of ruptured fibers by a counting process. In practice, however, these observations are actually decisions and as such require criteria. For each fiber, the observer must decide whether it was actually involved in the sheet fracture or simply exposed in fracture process, whether the fiber moved during fracture, and whether a matching fiber fragment can be located on the other side of the fracture zone and, if so, whether it had once been a part of the given fiber. These are subjective judgments and they can seriously affect the standard error of the measurement. The judgment error can be reduced through the use of photographs of the sheets taken prior to fracture, the sheets having been transparentized with xylene so that only the dyed fibers are visible. A fiber can be located before and after the sheet fracture and its role in the fracture can be readily assessed. This technique was employed in later work; however, because of the large number of measurements desired in these particular experiments, the more arbitrary but simpler method was used.

A one percent concentration of dyed fibers was used in these experiments; a higher concentration would have hindered the observation of individual fibers and a lower concentration would not have yielded as much information. (See Appendix III for procedure used.) A fiber extending into the fracture zone, determined by aligning the fractured sheet halves, was considered to have been involved in the fracture. If conceivably matching fiber fragments could be located on both sides of the fracture zone the fiber was said to have been ruptured; if not, it was pulled out.

The two halves of a fractured specimen were aligned with a straight edge and clamped between two lantern slides. The halves were positioned about one-eighth of an inch apart in order to define a zone of fracture. This zone of fracture was often difficult to define, particularly when the specimen had tended to delaminate during fracture, as frequently occurred during the Elmendorf tear test. In these cases, portions of the delaminated regions would overlap when the halves were separated one-eighth inch; consequently, they were excluded from observation. Delaminated regions that did not overlap also caused difficulty since well-defined fracture zone boundaries could not be established.

These slides were viewed through a binocular microscope at 20X magnification with transmitted light from an ultraviolet lamp. A yellow filter was mounted in front of the objective lens to filter the UV light and allow only the passage of the visible light from the tagged fibers which had been dyed with a UV fluorescing dye. This was done in a darkened room for maximum contrast. Pushbutton counters were used to record the total fibers observed and the ruptured fibers. Five specimens per sample were observed for samples fractured in tensile testing, Elmendorf tear (five sheet), Elmendorf tear (single sheet), and in-plane tear.

The fiber rupture frequencies encountered in this work were somewhat less than those reported in the literature (7,8) and consequently less than anticipated. Although the technique used in this phase was subject to considerable error due to the necessity of defining fiber involvement and passing judgment on the state of a fiber prior to sheet fracture, the differences were attributed to the use of classified pulps as opposed to whole pulps. Later work, where specimens were photographed before and after rupture, confirmed these differences. The rupture frequencies in the various modes of failure are shown in Appendix IV.

INTERPRETATION AND DISCUSSION

APPLICATION OF A TEAR EQUATION

In Phase II of the project proposal a need for a quantitative model is described. The purpose of such a model would be the prediction of sheet properties through knowledge of fiber properties and sheet structure. The starting point for such a model was the development of a tear equation under Group Project 2070. The assumptions of frictional pull-out and critical fiber length used by Wilder (9) in the development of this equation are very similar to those of Kane (10) in his development of a tear predictive equation. While Wilder's equation was, admittedly, subject to limitations such as the assumption of equal load distribution from fiber to fiber, and neglect of angular orientation of fibers, and while there may be some question as to the validity of treating fiber pull-out as a length-dependent frictional force phenomenon and of the existence of a critical length, this model represents an attempt to describe a quantitative relationship between tear and fiber properties.

Wilder's tear equation was presented in Project 2070, Report Eleven in the following form:

$$\frac{ty}{a_r(A + L_c^3C)} = k_3 \frac{yz(1-B-L_cC)}{a_r(A + L_c^3C)} + k_5 \qquad (1)$$

where: t = tear factor

y = pulp yield, fraction of original

a_r = relative bonded area

L_c = "critical" fiber length. All fibers shorter than this length are pulled out during tear

z = zero span tensile strength

k_3, k_5 = constants

and $\underline{A}$, $\underline{B}$, and $\underline{C}$ are the integrals:

$$A = \int_{0}^{L_c} L^2 \cdot Y(L) \cdot dL$$

$$B = \int_{0}^{L_c} Y(L) \cdot dL$$

$$C = \int_{L_c}^{\infty} \frac{1}{L} \cdot Y(L) \cdot dL$$

in which $\underline{L}$ is fiber length and $\underline{Y(L) \cdot dL}$ represents the fraction of all fibers having a length between $\underline{L}$ and $\underline{L} + \underline{dL}$.

The integral $\underline{B}$ is the summation of fibers of length less than $\underline{L_c}$ (all of which are assumed to be pulled from the sheet) and $\underline{C}$ is the summation for fibers longer than $\underline{L_c}$ that are pulled from the sheet where the factors $\underline{L_c}/\underline{L}$ is the fraction of fibers of length $\underline{L}$ pulled from the sheet. The term $(1-\underline{B}-\underline{L_c}\underline{C})$ represents the fibers ruptured. The integral $\underline{A}$ is a factor relating pull-out lengths of fibers less than $\underline{L_c}$ to their pull-out energy contribution.

The critical fiber length was assumed to be related to fiber strength and bonded area in the following manner:

$$L_c = k_6 \left(\frac{z}{a_r}\right) \quad . \tag{2}$$

The pulp yield, $\underline{y}$, was used by Wilder as an index of the number of fibers involved in the tear, the number of fibers at constant basis weight being inversely proportional to the yield. In applying Equation (1) to this work it was felt that the calculated number of fibers crossing a one-millimeter line would be a more direct indication of the number of fibers involved in the tear. This value was calculated from the equation:

$$N_f = 100 \cdot \frac{W}{C} \left(\frac{2}{\pi}\right) \tag{3}$$

where $\underline{N_f}$ = the number of fibers crossing a one-millimeter line

$\underline{W}$ = basis weight in g./m.^{2}

$\underline{C}$ = coarseness in mg./100 m.

$2/\pi$ = angular distribution function for random orientation

Wilder's original equation was modified to become:

$$\frac{t}{a_r N_f (A + L_c{}^3 C)} = k_3 \frac{z\ (1\text{-}B\text{-}L_c C)}{a_r N_f (A + L_c{}^3 C)} + k_5 \quad . \tag{4}$$

It is difficult to present a qualitative discussion of Equation (4) since the interrelationships of some of the variables are not straightforward. The left-hand side of the equation is proportional to the fiber rupture frequency, $(1\text{-}\underline{B}\text{-}\underline{L_c C})$ and seems to be proportional to $\underline{z}$ and, in turn, is a limit of the integrals $\underline{A}$, $\underline{B}$, and $\underline{C}$. This issue is confounded by the fact that the left-hand side of the equation has no physical significance unless the denominator of $\underline{t}$ is transposed to the right-hand side which places the equation in a nonlinear form.

The equation was evaluated, quantitatively, with the same procedure used by Wilder. The constants $\underline{k_3}$, $\underline{k_5}$, and $\underline{k_6}$ were determined by assuming values of $\underline{k_6}$ and calculating $\underline{k_3}$ and $\underline{k_5}$ as a slope and intercept of Equation (4). The fit of the data for an assumed $\underline{k_6}$ was judged from the correlation coefficient for a least squares fit of the data to a straight line. The Institute's computer facilities were utilized in this work with a program that would make repeated calculations for a range of assumed values of $\underline{k_6}$ and print out the values of $\underline{k_3}$ and $\underline{k_5}$ and the correlation coefficient for a given set of data (obtained by varying wet pressing for each of the nine pulp samples). The maximum correlation was then used to select the "best" values of the constants. The integrals $\underline{A}$, $\underline{B}$, and $\underline{C}$ were computed by numerical summation of the fiber length distribution data setting $\underline{dL}$

equal to a length distribution interval and $\underline{Y}(\underline{L})$ equal to the fraction of the fiber population in an interval $\underline{dL}$.

One of the deficiencies that became apparent as the program was run was the number of modes occurring in the correlation coefficients calculated over a range of assumed constants. A scan of one hundred assumed values of $\underline{k}_6$ selected to result in critical fiber lengths ranging from less than 0.1 mm. to the maximum length observed in the population generally produced several "best" values, equally acceptable, on this basis, that would predict fiber rupture frequencies ranging from near zero to very high for the same set of data. This made it necessary to pick the best of the "best" values by some other criterion. Constants were selected that resulted in rupture frequencies most consistent with observed values.

The five-ply Elmendorf and the in-plane tear data were processed by the computer program. Although, as has been mentioned, the single-ply Elmendorf test seems to be the more desirable test, the five-ply data were used with the possibility in mind of a comparison with the preceding Project 2070 analysis. The best fit constants for these two tests are shown in Tables I and II. Computations resulting in negative valued constants were disregarded even though, in some cases, correlations were high since they would not be logically consistent with the tear equation analysis. The constants shown in these tables represent peak correlations having correlation coefficients within five points of the maximum observed correlation coefficient.

The calculated fiber pull-out frequencies were compared with the experimentally observed pull-out frequencies. The calculated frequencies best agreeing with the observed frequencies are given in Tables III and IV for the Elmendorf and in-plane tearing modes, respectively. Also shown are the corresponding tear equation constants and critical fiber lengths.

TABLE I

BEST FIT VALUES OF TEAR EQUATION CONSTANTS FOR FIVE-PLY ELMENDORF TEAR

White oak:							
Unbeaten	k_3	0.0570	0.0719	0.0764	0.0954		
	k_5	0.0462	0.1331	0.4847	0.5446		
	k_6	0.00008	0.00010	0.00012	0.00014		
	Correlation coefficient	0.940	0.938	0.950	0.928		
10-Min. beating	k_3	0.1742	0.2670	0.2694	1.052		
	k_5	0.0722	0.7473	0.8365	0.8556		
	k_6	0.00015	0.00025	0.00030	0.00048		
	Correlation coefficient	0.961	0.933	0.938	0.938		
20-Min. beating	k_3	0.2707	0.2728				
	k_5	1.543	1.628				
	k_6	0.00027	0.00030				
	Correlation coefficient	0.832	0.822				
Aspen:							
Unbeaten	k_3	0.0729	0.0997	0.1362			
	k_5	1.290	1.231	0.8717			
	k_6	0.00011	0.00017	0.00022			
	Correlation coefficient	0.993	0.986	0.990			
10-Min. beating	k_3				0.5480		
	k_5				0.9993		
	k_6				0.00048		
	Correlation coefficient				0.878		
20-Min. beating	k_3	0.0637	0.0686	0.0830	0.1156		
	k_5	155.4	27.24	5.270	2.305		
	k_6	0.00001	0.00005	0.00013	0.00022		
	Correlation coefficient	0.991	0.992	0.992	0.967		
2406-A:							
5-Min. beating	k_3	0.2669	0.2965	0.3324	0.3734	0.3979	0.4652
	k_5	8.157	5.583	4.577	3.873	3.469	2.877
	k_6	0.00012	0.00015	0.00021	0.00024	0.00030	0.00037
	Correlation coefficient	0.990	0.987	0.973	0.973	0.954	0.956
20-Min. beating	k_3	0.1490	0.1562	0.1778	0.1783		
	k_5	14.918	660.9	34.25	13.75		
	k_6	0.00001	0.00003	0.00010	0.00017		
	Correlation coefficient	0.921	0.981	0.978	0.931		
40-Min. beating	k_3	0.1121	0.1365				
	k_5	645.2	27.21				
	k_6	0.00004	0.00013				
	Correlation coefficient	0.873	0.898				

TABLE II

BEST FIT VALUES OF TEAR EQUATION CONSTANTS FOR IN-PLANE TEAR

White oak:

Unbeaten	k_3	0.2914	0.3771				
	k_5	0.3157	0.9271				
	k_6	0.00012	0.00014				
Correlation coefficient		0.948	0.940				
10-Min. beating	k_3	0.4919	0.9595	0.7237	0.7081		
	k_5	3.551	1.247	2.809	2.961		
	k_6	0.00015	0.00021	0.00025	0.00030		
Correlation coefficient		0.967	0.903	0.933	0.924		
20-Min. beating	k_3	1.354	0.9618				
	k_5	3.512	5.068				
	k_6	0.00018	0.00027				
Correlation coefficient		0.820	0.869				

Aspen:

Unbeaten	k_3	0.2742	0.3774	0.5199			
	k_5	1.204	2.582	1.952			
	k_6	0.00012	0.00017	0.00022			
Correlation coefficient		0.988	0.985	0.962			
10-Min. beating	k_3	0.4553	0.7932	0.6188	0.6558		
	k_5	4.559	1.900	3.904	3.734		
	k_6	0.00015	0.00023	0.00035	0.00044		
Correlation coefficient		0.991	0.987	0.976	0.973		
20-Min. beating	k_3	0.3138	0.3290	0.5751	0.4940	0.5403	
	k_5	4.953	1.422	6.625	6.752	6.191	
	k_6	0.00007	0.00009	0.00023	0.00027	0.00030	
Correlation coefficient		0.9998	0.9998	0.993	0.996	0.997	

2406-A:

5-Min. beating	k_3	0.5459	0.6426	0.8450	0.9456		
	k_5	14.20	10.39	6.518	5.161		
	k_6	0.00012	0.00017	0.00035	0.00049		
Correlation coefficient		0.995	0.993	0.978	0.950		
20-Min. beating	k_3	0.5957	0.6878	0.8534	0.9895		
	k_5	106.9	56.00	25.99	14.22		
	k_6	0.00004	0.00009	0.00014	0.00030		
Correlation coefficient		0.992	0.992	0.986	0.966		
40-Min. beating	k_3	0.6878	0.7300	0.9415	0.7888	0.9279	1.048
	k_5	46.55	52.18	23.23	17.46	14.39	12.40
	k_6	0.00008	0.00013	0.00021	0.00035	0.00042	0.00052
Correlation coefficient		0.990	0.992	0.983	0.951	0.991	0.951

TABLE III

COMPARISON OF CALCULATED AND EXPERIMENTAL PULL-OUT FREQUENCIES FOR FIVE-PLY ELMENDORF TEAR

	Relative Bonded Area, %	k_6	Fiber Pull-Out Fequency, % Calculated	Fiber Pull-Out Fequency, % Experimental	Critical Fiber Length, mm.
White oak:					
Unbeaten	12	0.00012	83	90	0.69
	7		92	93	1.18
	16		70	86	0.59
	13		80	87	0.73
	16		76	86	0.64
	20		58	83	0.51
10-Min. beating	17	0.00030	98	92	1.34
	19		94	92	1.19
	21		96	88	1.13
	24		88	88	1.02
	24		86	87	1.05
	25		87	89	1.04
20-Min. beating	16	0.00027	98	93	1.36
	18		93	92	1.18
	19		88	91	1.25
	17		85	92	1.29
	20		95	91	1.15
	19		89	85	1.24
Aspen:					
Unbeaten	18	0.00022	94	97	0.86
	19		92	96	0.82
	17		82	94	1.06
	23		91	94	0.82
	28		83	92	0.72
	24		78	92	0.60
10-Min. beating	23	0.00048	100	96	1.51
	28		92	96	1.28
	29		99	96	1.37
	33		93	97	1.25
	37		96	96	1.18
	38		98	94	1.12
20-Min. beating	20	0.00022	86	93	0.90
	28		84	95	0.67
	36		77	93	0.57
	35		71	92	0.58
	41		71	90	0.51
	49		67	91	0.41

TABLE III (Continued)

COMPARISON OF CALCULATED AND EXPERIMENTAL PULL-OUT FREQUENCIES
FOR FIVE-PLY ELMENDORF TEAR

	Relative Bonded Area, %	k_6	Fiber Pull-Out Frequency, %		Critical Fiber Length, mm.
			Calculated	Experimental	
2406-A:					
5-Min. beating	17	0.00037	84	92	1.39
	18		87	91	1.50
	20		84	93	1.40
	17		90	93	1.74
	23		77	91	1.28
	31		71	91	0.92
20-Min. beating	18	0.00017	50	95	0.77
	19		68	96	0.74
	23		64	94	0.65
	15		76	97	1.02
	24		61	96	0.61
	29		54	95	0.53
40-Min. beating	26	0.00013	61	95	0.44
	34		44	96	0.34
	35		44	94	0.34
	33		51	92	0.38
	36		40	92	0.32
	37		43	92	0.33

TABLE IV

COMPARISON OF CALCULATED AND EXPERIMENTAL PULL-OUT FREQUENCIES FOR IN-PLANE TEAR

	Relative Bonded Area, %	k_6	Fiber Pull-Out Frequency, %		Critical Fiber Length, mm.
			Calculated	Experimental	
White oak:					
Unbeaten	12	0.00014	89	86	0.80
	7		97	91	1.38
	16		83	82	0.69
	13		93	80	0.85
	16		82	82	0.74
	20		70	82	0.59
10-Min. beating	17	0.00025	97	92	1.12
	19		89	86	0.99
	21		91	87	0.94
	24		84	85	0.85
	24		83	84	0.87
	25		84	85	0.86
20-Min. beating	16	0.00018	91	88	0.91
	18		84	89	0.78
	19		85	86	0.83
	17		85	90	0.86
	20		83	82	0.76
	19		85	81	0.82
Aspen:					
Unbeaten	18	0.00022	94	92	0.87
	19		92	92	0.82
	17		82	88	1.05
	23		91	86	0.82
	28		83	82	0.72
	24		78	83	0.60
10-Min. beating	23	0.00035	99	96	1.10
	28		95	95	0.93
	29		91	94	1.00
	33		95	95	0.91
	37		83	88	0.86
	38		85	92	0.82
20-Min. beating	20	0.00030	93	94	1.22
	28		95	93	0.92
	36		88	94	0.77
	35		88	92	0.78
	41		85	88	0.70
	49		76	89	0.56

TABLE IV (Continued)

COMPARISON OF CALCULATED AND EXPERIMENTAL PULL-OUT FREQUENCIES
FOR IN-PLANE TEAR

	Relative Bonded Area, %	k_6	Fiber Pull-Out Frequency, % Calculated	Experimental	Critical Fiber Length, mm.
2406-A: 5-Min. beating	17	0.00049	90	92	1.84
	18		93	94	1.99
	20		90	93	1.86
	17		96	90	2.30
	23		87	91	1.69
	31		78	88	1.22
20-Min. beating	18	0.00030	87	93	1.36
	19		87	97	1.31
	23		82	93	1.15
	15		92	95	1.80
	24		75	96	1.08
	29		78	96	0.93
40-Min. beating	26	0.00052	96	94	1.75
	34		93	93	1.36
	35		93	92	1.37
	33		94	90	1.51
	36		86	93	1.29
	37		93	91	1.34

Immediately apparent from this work are two points of criticism that can be lodged against this equation, or any other analysis based upon the fit of data to an equation containing a number of measured quantities each subject to error. First, the equation tends to have a smoothing effect as illustrated by the high degrees of correlation observed using relative bonded area data subject to considerable error and the number of modes of high correlation from which conflicting frequencies of fiber pull-out could be calculated. Second, constants giving agreement with experimental rupture frequency observations could not be related to any observed pulp properties and therefore provided no means of ascertaining specific constants for a given pulp without reference to experimental rupture frequency data.

The concept of critical fiber length has questionable validity inasmuch as it presumes that a fiber is pulled from a sheet with bond rupture occurring progressively in a frictionlike manner. To the contrary it has been observed in dyed fiber rupture experiments where photographs were used to identify fibers prior to rupture, that many times a fiber would rupture at its tip sometimes leaving a fragment not much longer than its width.

EXTENSIONAL STIFFNESS AND SPECIFIC SURFACE

While the use of unbonded surface area measurements seems to be superior to the scattering coefficient – tensile strength method of determining relative bonded area, the fact remains that when the bonded areas are low the relative bonded area calculations are subject to considerable error. This is because the calculations are dependent upon small differences between experimentally determined values. It would seem that if the surface area measurements could be plotted against another experimental measurement in such a manner as to produce a well-defined curve the data could be "smoothed," thereby producing better estimates of relative bonded area.

Working with these data, Brezinski observed that a semilogarithmic plot of unbonded surface area versus extensional stiffness described a straight line with a zero intercept very near to the experimentally determined unbonded fiber area. Plots for the three pulp species are shown in Fig. 1. Surprisingly, all three pulps describe lines of essentially equal slope indicating that the extensional stiffness is dependent upon relative bonded area and independent of pulp species. This means that the extensional stiffness is a property of the fiber network and not the fibers themselves. Possibly this independence would break down at high levels of bonding and it may be influenced by the presence of fines, which were excluded in this work. Should this relationship be confirmed or found to be accurate within the levels of bonding considered here it would provide an accurate and simplified measure of relative bonded area. When sheet strength properties were plotted against relative bonded area the smoothed values taken from Fig. 1 reduced the scatter of these plots; however, good correlations between relative bonded area and fiber rupture frequencies were not obtained due to the low magnitude and comparatively large errors in the latter.

SHEET STRENGTH PROPERTIES

For the purpose of graphical representation the strength data were plotted against density and, for cross-reference, density was plotted against relative bonded area for these pulps. The choice of density as an independent variable was an arbitrary one based solely on the fact that the data defined smooth curves with little scatter. Plots of tensile breaking length, in-plane tear energy, and single-ply Elmendorf tear factor versus density are shown in Fig. 2, 3, and 4 respectively, while density is plotted against relative bonded area in Fig. 5.

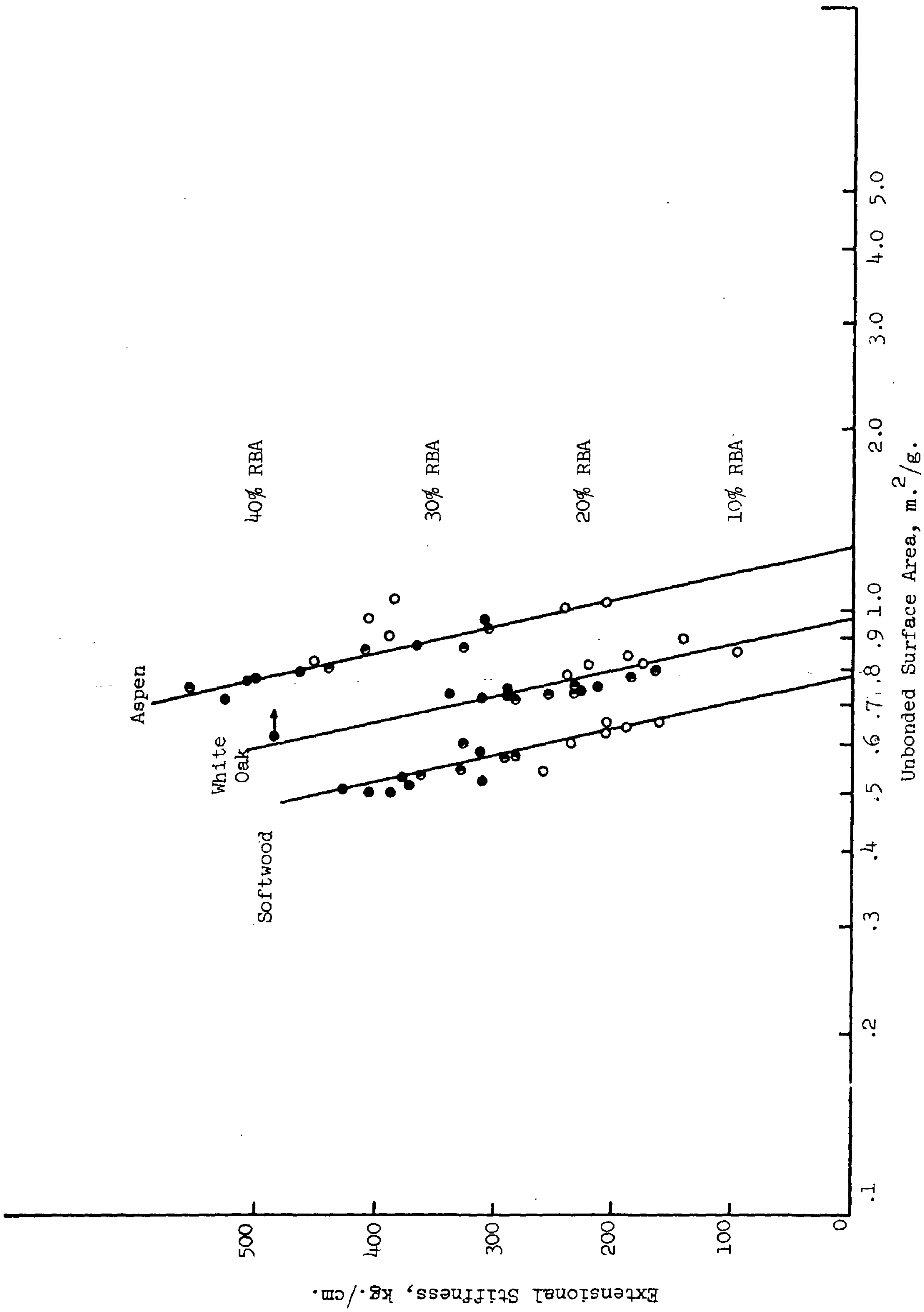

Figure 1. Relation of Extensional Stiffness to Unbonded Surface Area

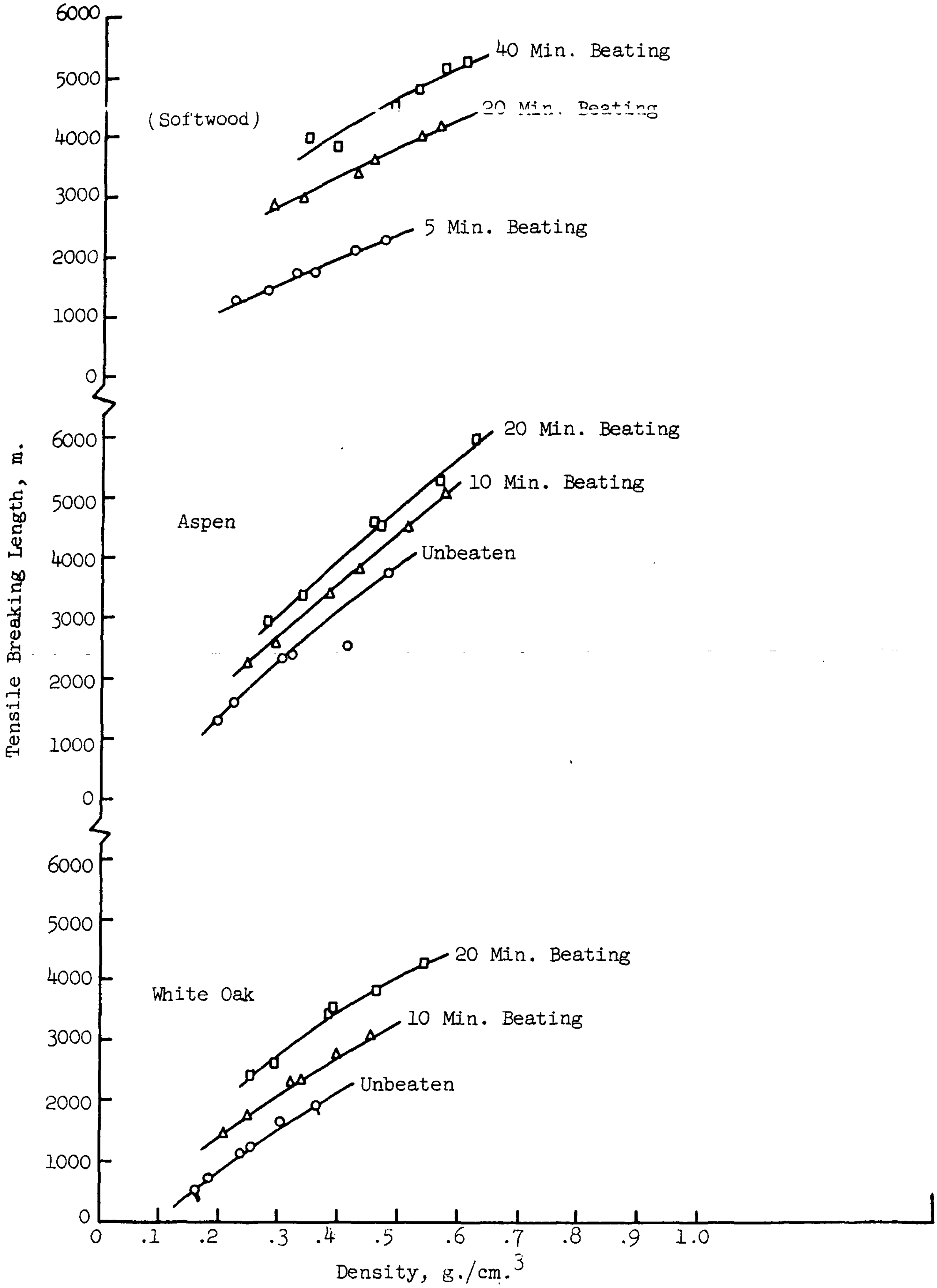

Figure 2. Relation of Tensile Breaking Length to Density

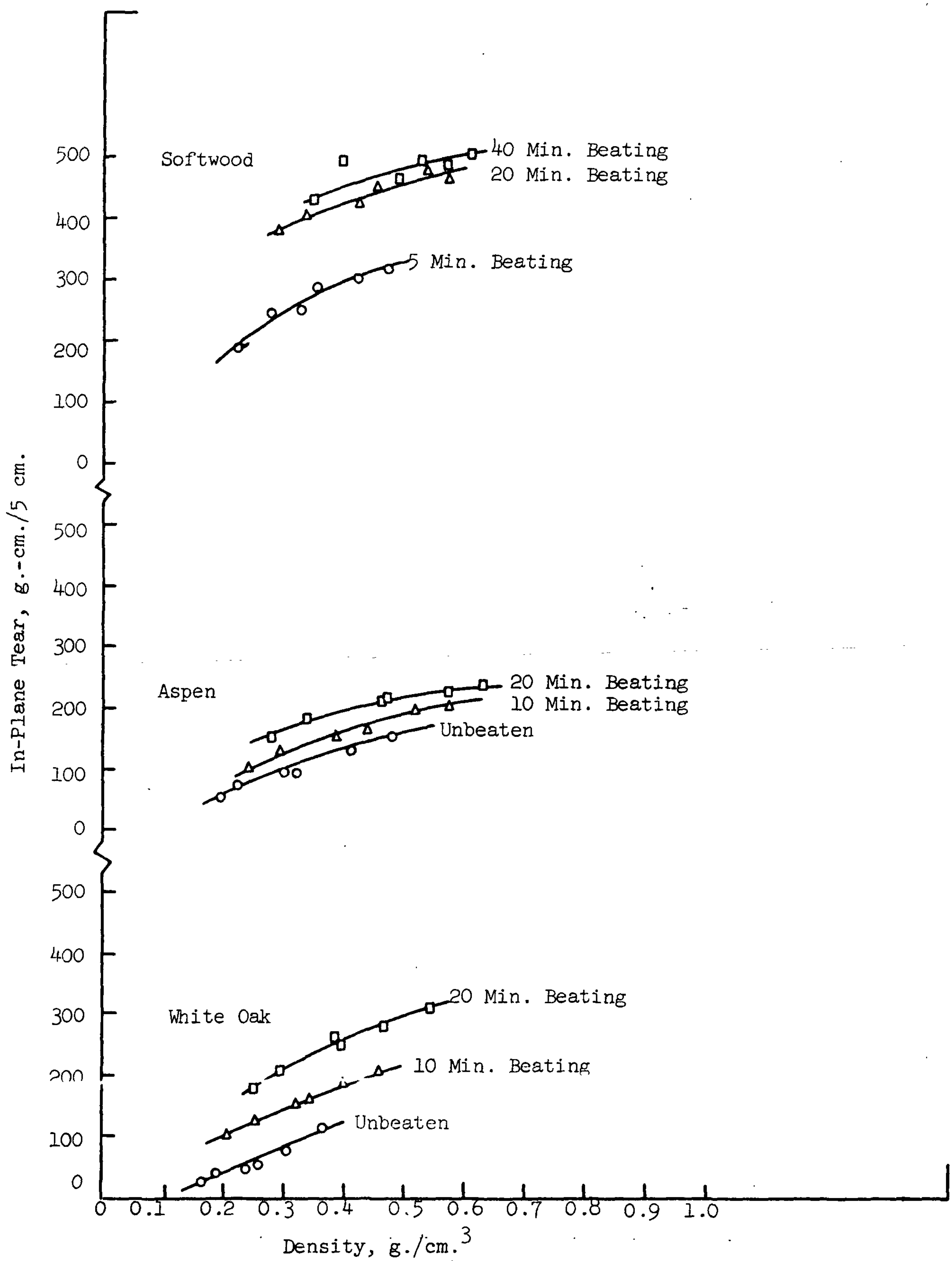

Figure 3. Relation of In-Plane Tear to Density

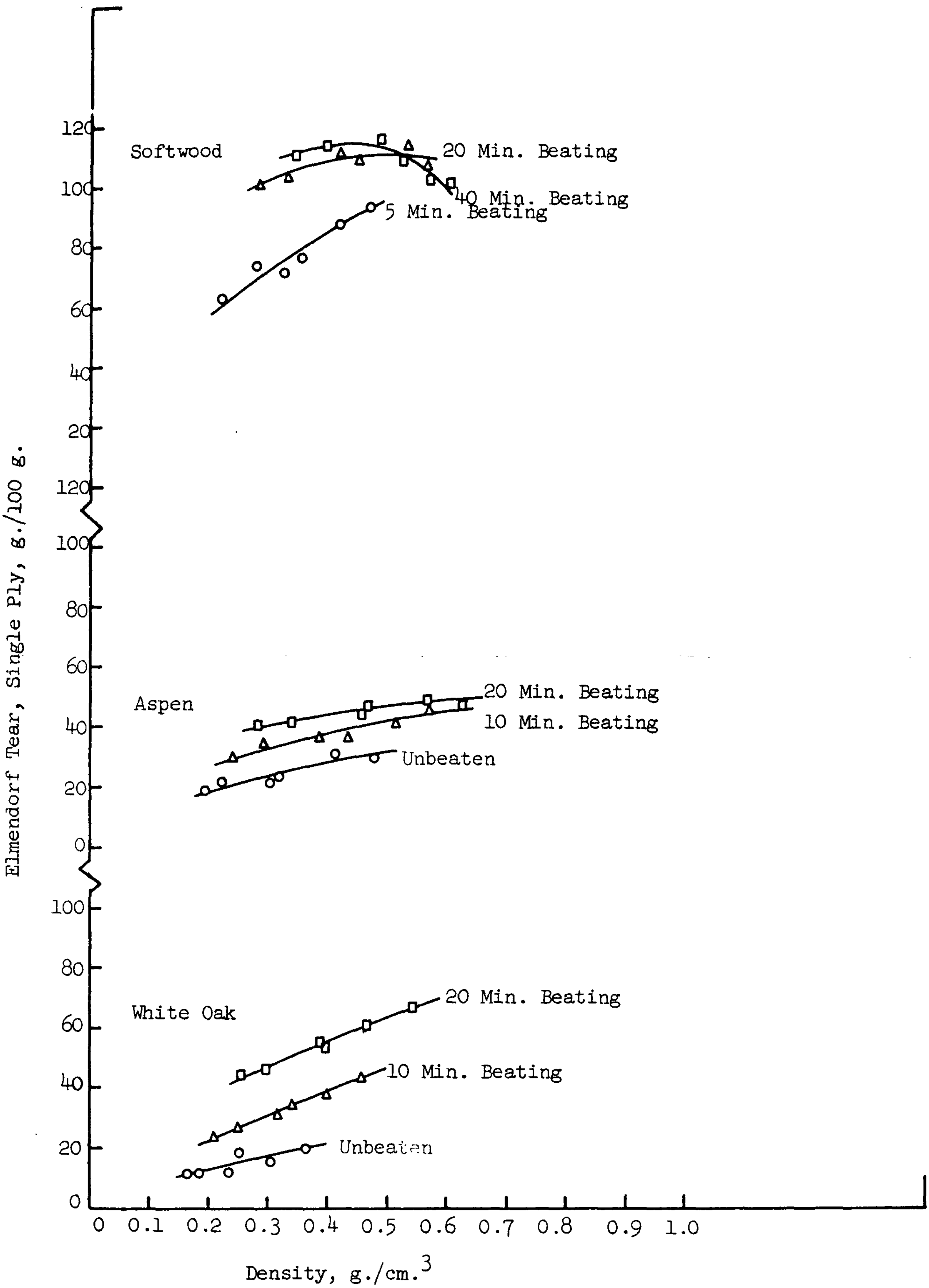

Figure 4. Relation of Elmendorf Tear to Density

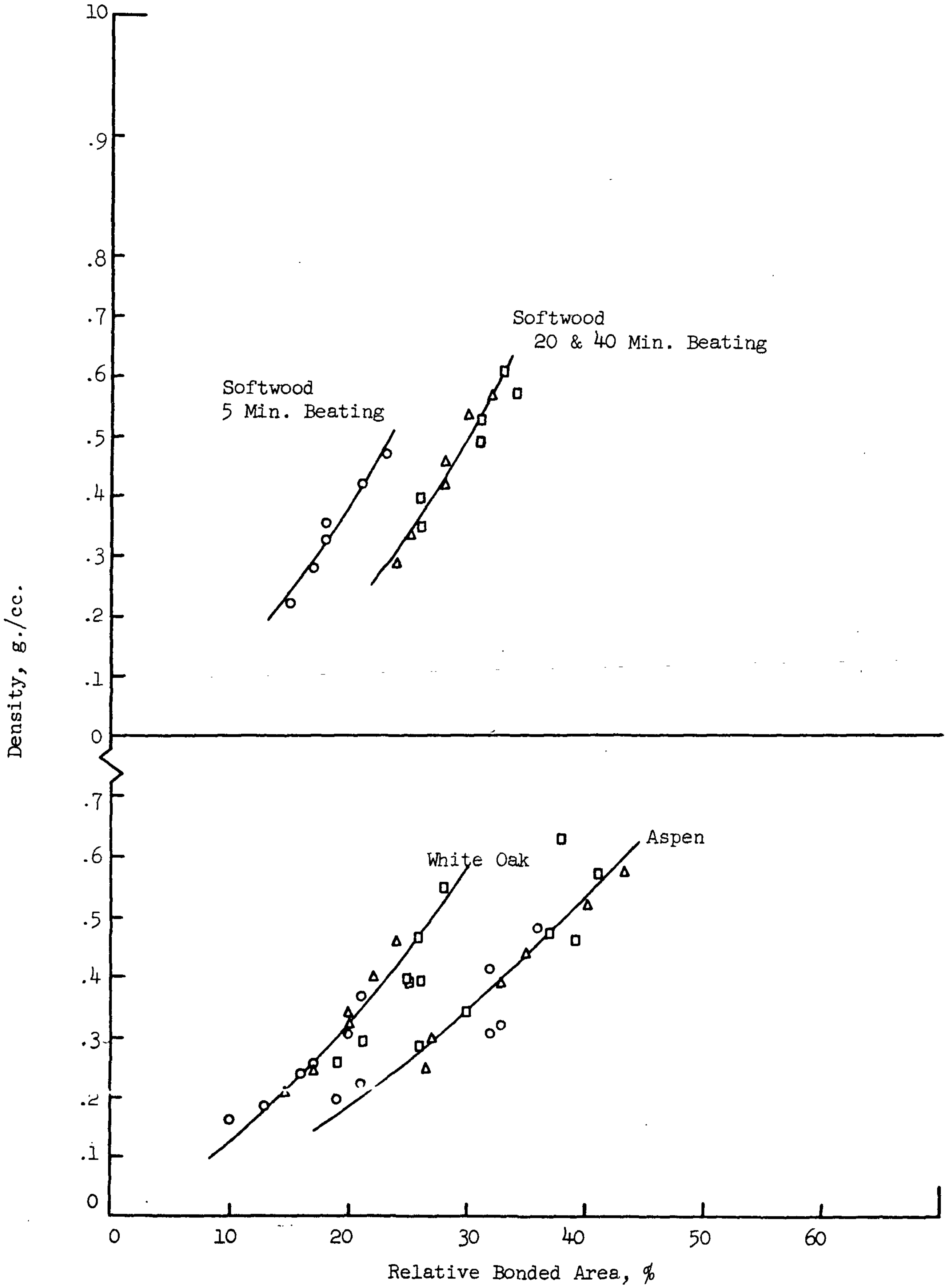

Figure 5. Relation of Density to Relative Bonded Area

In the case of the hardwoods, beating was a parameter along which the sheet strength properties increased with density. This was also the case with the softwood; however, the effect of beating was more pronounced and the Elmendorf tear exhibited a maximum at the higher beating intervals. The relationship between density and relative bonded area showed no discrimination in regard to beating in so far as the hardwoods were concerned while two distinctly different curves were obtained for the mildly beaten and the more highly beaten softwoods.

TEARING STRENGTH AND FIBER LENGTH

Considering the case of tear properties there are two points which can provide for an intuitive relationship between tearing strength and fiber dimensions: (1) tear is a network failure phenomenon, and (2) the structure of a network is related to the dimensions of its elements. It can be postulated that tearing strength is a function of average fiber length and pulp species can be compared on this basis. However, there are other properties that can exert an overriding influence on tearing strength; this is apparent from the typical beating study where it is commonly observed that the average fiber length is shortened while tearing strength increases. To account for these competing influences a secondary basis for comparison is necessary. Such a basis must be sensitive to the same influences as tearing strength with the exception of fiber length. This problem was discussed and led to the development of a tear equation in the latter phases of Project 2070 because no meaningful basis for comparison could be found.

Comparison of tearing strengths (Elmendorf) at constant tensile strength is not a new approach and it was rejected as a basis of comparison in Project 2070. Nevertheless, in the work reported here it has been noted that the tensile strength development for all three species approached values of nearly equal magnitude. On the other hand, tearing strength development reached significantly different levels.

Although both strength properties are considered to be dependent upon fiber length, it appears that tearing strength, in this case, has a comparatively greater dependency upon fiber length — possibly this difference has been enhanced by the removal of fines from the experimental pulps. Fines not only make an obscure contribution to the network structure, they occlude the characterization of fiber length. In this situation there seems to be reasonably good cause for a comparison of tearing strengths at equal tensile strengths.

Tear energy was plotted against the tensile breaking load for the three pulps as shown in Fig. 6, 7, and 8. From these plots in-plane tear energies were determined at tensile breaking loads of 1000, 2000, and 3000 g./cm. and plotted against average fiber length in Fig. 9. Although few data points are available, a relationship between average fiber length and in-plane tear is apparent; a similar relationship is also apparent with the Elmendorf tear (single ply), but the reduction in tear energy for the softwood at the higher tensile strength indicates that the effect of fiber shortening has become dominant in regard to the beating process.

In addition to the dependency of tear energy on length, the response of average fiber length to beating is evident in Fig. 9 with the order in which pulps are shortened with beating being softwood > aspen > white oak. Interestingly, this order reflects the coarseness values shown in Table V of Appendix I; however, it would be premature to attempt to define any relationship between coarseness and this beating response.

The implications of these data are encouraging in that they point to the possibility of defining relationships between tearing strength (in-plane) and fiber length that would not be specific to a single fiber species. Further work utilizing classified fiber fractions in which average fiber lengths are more significant representations could affirm and clarify this evident dependency.

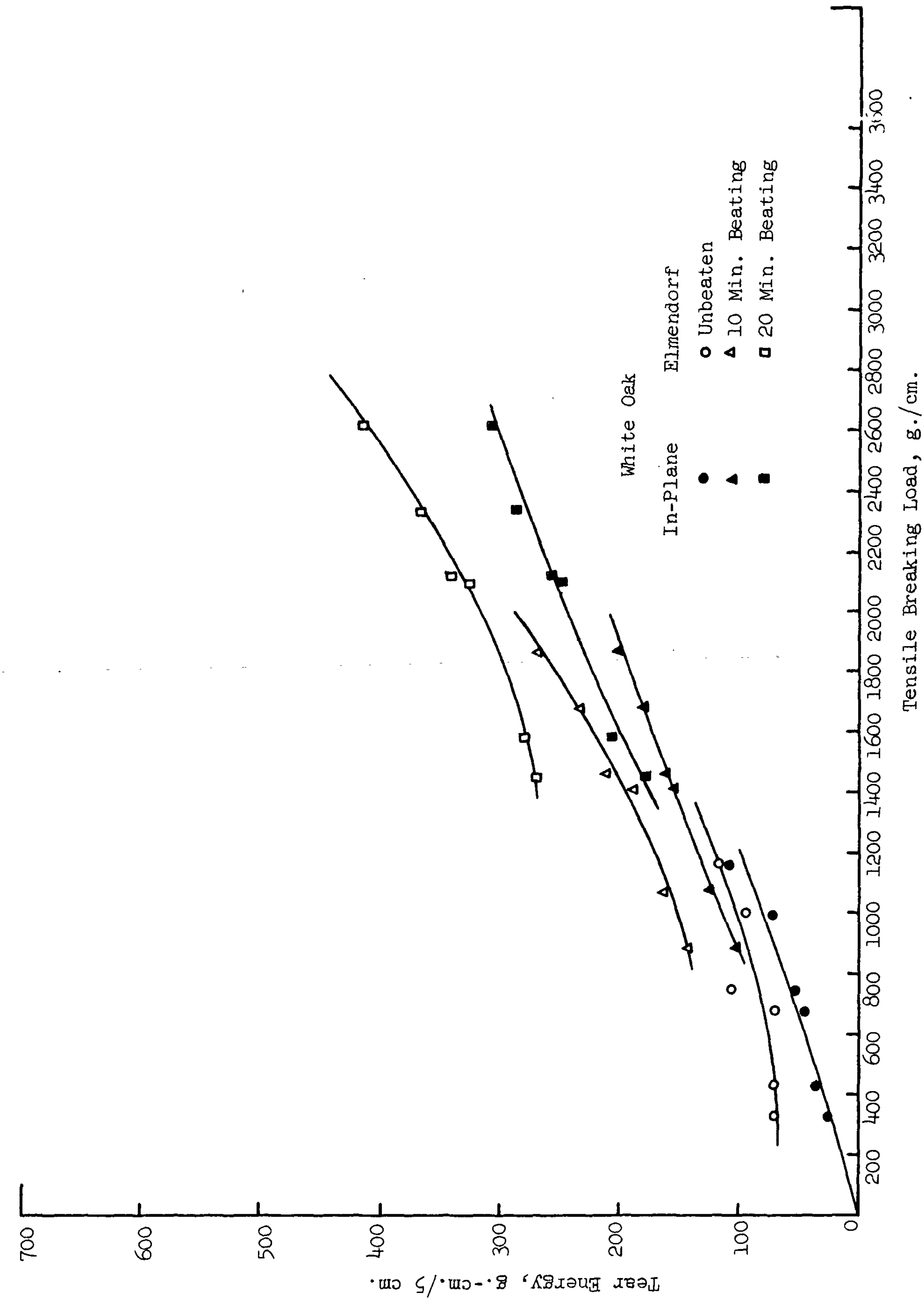

Figure 6. Relation of Tear Energy to Tensile Breaking Load for White Oak

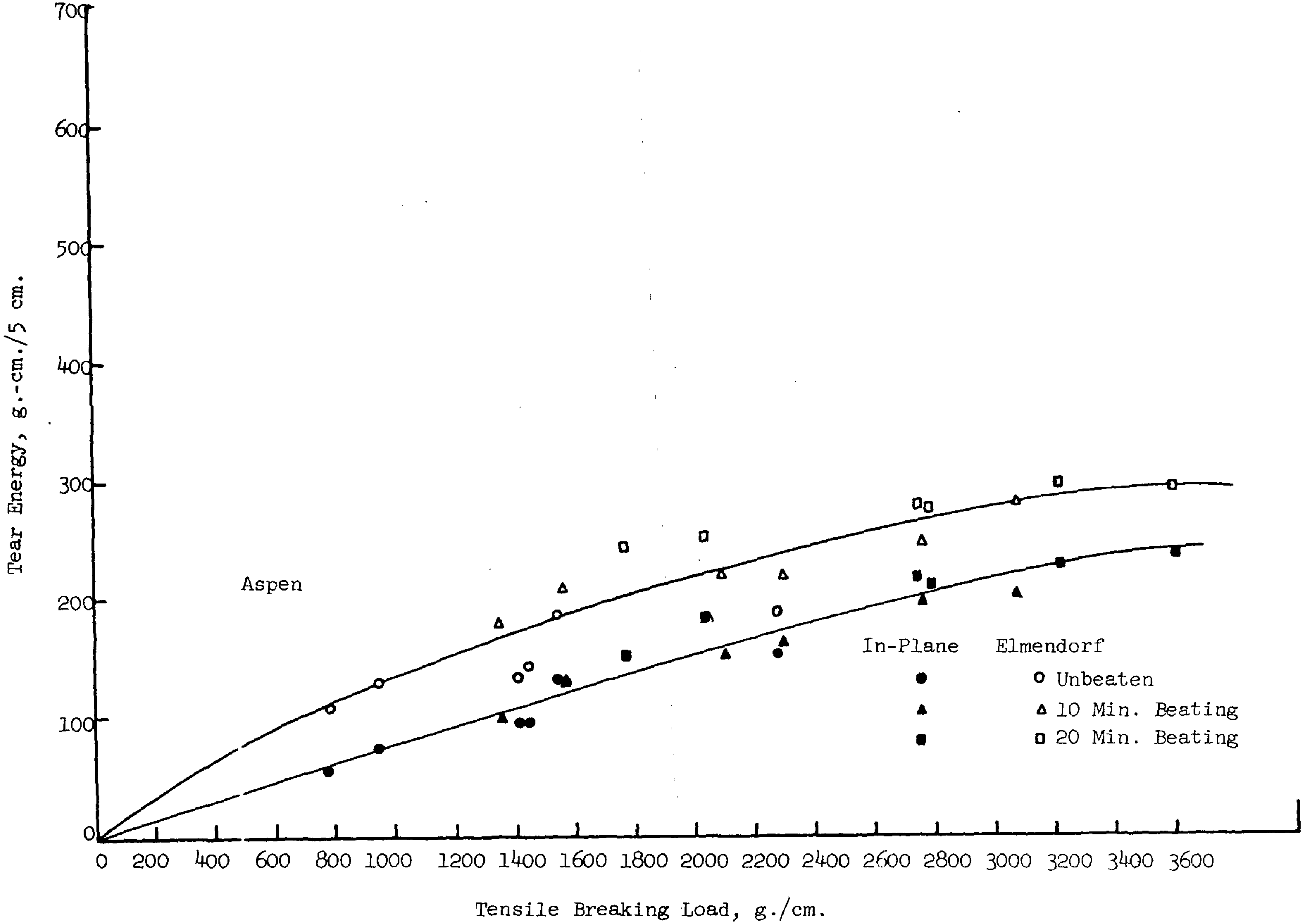

Figure 7. Relation of Tear Energy to Tensile Breaking Load for Aspen

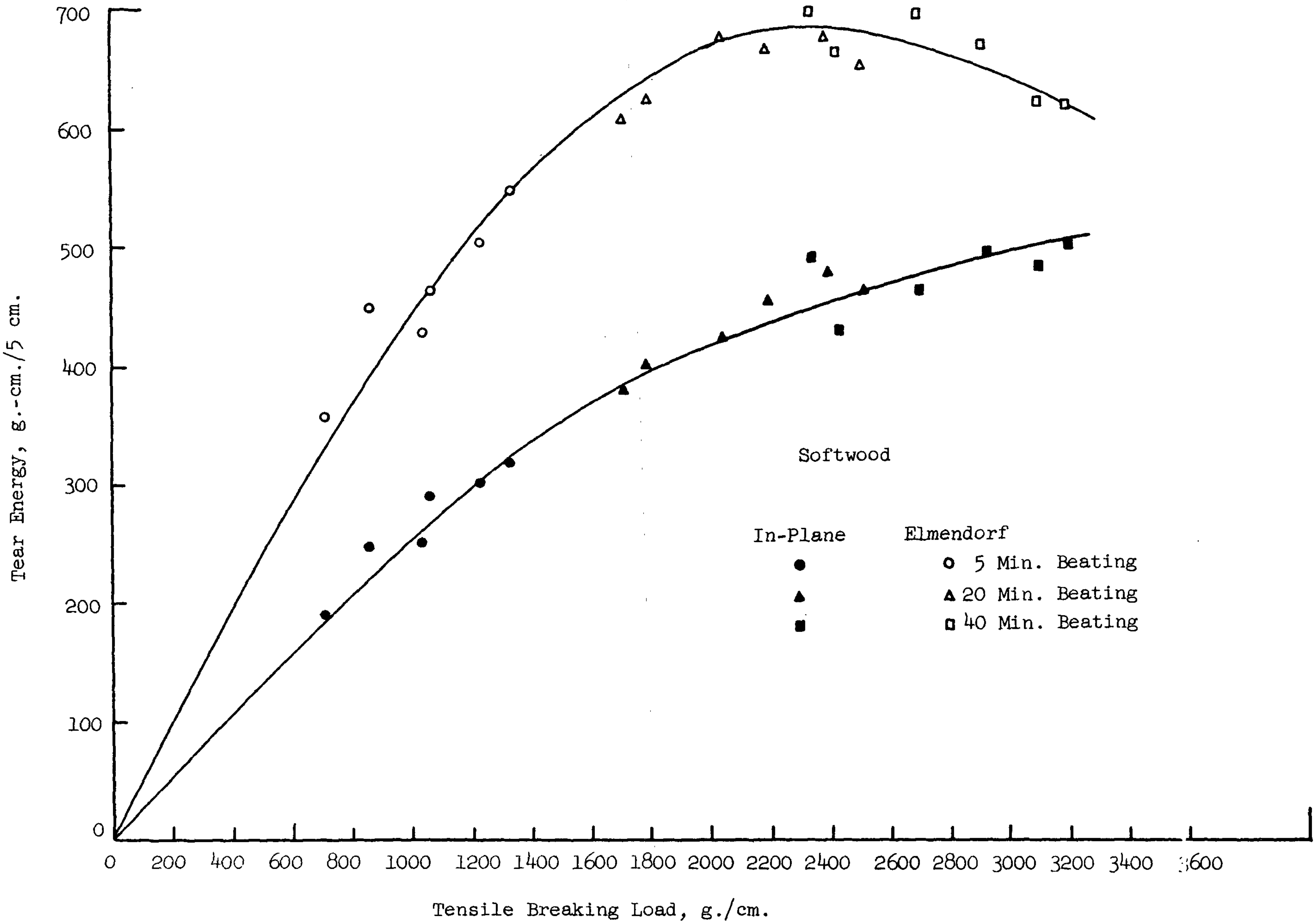

Figure 8. Relation of Tear Energy to Tensile Breaking Load for Softwood

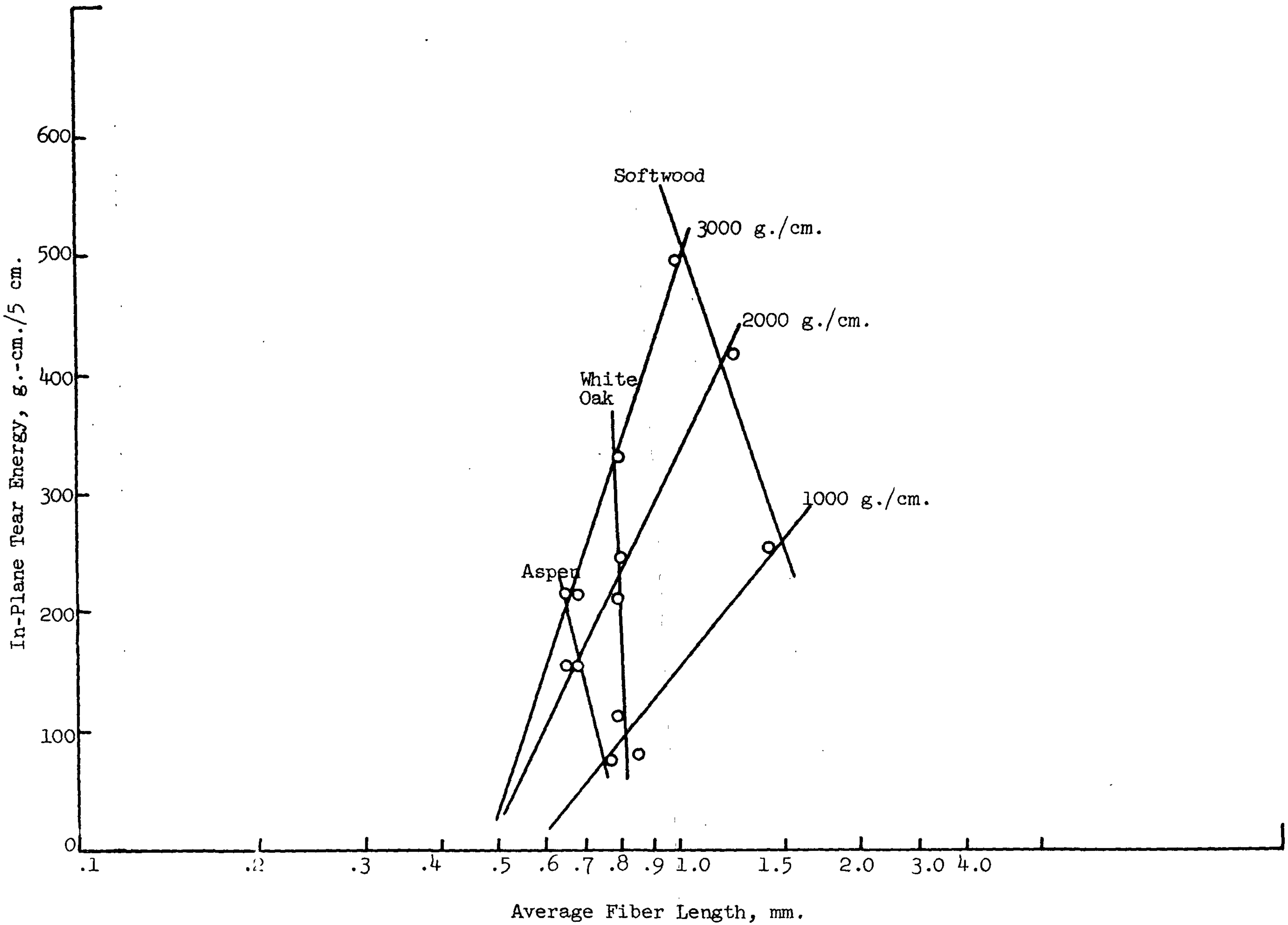

Figure 9. Relation of In-Plane Tear to Fiber Length and Tensile

RUPTURE FREQUENCY

At equivalent breaking lengths the order of rupture frequency was white oak > aspen > softwood. This was also the case for in-plane and Elmendorf tear failure at equivalent strengths. At equal degrees of bonding the order of rupture frequency was white oak > softwood > aspen for tensile and Elmendorf tear failure while the in-plane tear failure order was white oak > aspen > softwood with the aspen and softwood frequencies becoming nearly equal at the higher stages of beating. It would appear from this that the white oak was the weaker of the pulps, as was indicated by the zero-span measurements. The zero-span strengths of the aspen and the softwood were very nearly the same and, it is apparent from Fig. 5 that the relative bonded area of the aspen sheets increased more rapidly with density than the others. From this it can be seen that the aspen fibers are essentially as strong (by the zero-span test) and more flexible than the softwood, yet at equal degrees of bonding and at equal densities it produced sheets inferior in tear strength to both the longer fibered white oak and softwood sheets. Again it seems that it is the fiber length that is paramount in sheet strength development.

CONCLUSIONS

The intent of this work was an evaluation of the tear equation evolved by Wilder in the closing phases of Project 2070. The equation showed promise on the basis of Wilder's partial evaluation; however, the application of the equation to the work reported here has not been successful and has indicated that the application of such an equation may be premature. The derivation of the equation required some knowingly tenuous assumptions based upon incomplete understanding of the tearing process. For example, assumptions were necessary as to the nature of the force distribution along a fiber during tear and effect of fiber orientation to the tear axis pertaining to the impressed forces and involvement in the tear process. These assumptions were discussed at the time the equation was derived; and the apparent fit to experimental data, at that time, seemed to exonerate them. The failure of the equation in this later work has come not because of an inability to obtain good fit of the data to the equation but the inability of the equation to predict fiber rupture frequencies. Primarily this is due to the smoothing effect of the equation resulting in several peak correlations within a range of assumed constants and allowing one to pick at will the rupture frequencies for a given set of data. In addition, the lack of a relationship between the equation constants and observed pulp properties necessitated the assumed-constant trial and error approach.

A relationship between average fiber length and in-plane tear has been shown when the tear data are plotted at constant levels of tensile strength. This relationship becomes evident because of the comparative insensitivity of the tensile strength to fiber length. This situation may be specific to this work inasmuch as the pulps were classified. Nevertheless the approach is encouraging to further work.

One observation, especially worth noting, has been the apparent relationship between extensional stiffness and specific surface. Extrapolation of the extensional stiffness versus specific surface curves to zero extensional stiffness gives intercept values nearly equal to the experimental specific surface area values for unbonded fibers. Data for all three pulp species, white oak, aspen, and western softwood, seemed to define equally sloped straight lines in a semilogarithmic plot. Thus, it would appear that the extensional stiffness is solely dependent upon relative bonded area and independent of pulp species. This may be a fortuitous observation or, as suggested in the preceding case, specific to the conditions of this work. However, should it be confirmed by further work, even if limited to fines-free pulps, it could provide a simple yet accurate measure of relative bonded area.

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APPENDIX I

PREPARATION AND CHARACTERIZATION OF PULPS

Pulps were cooked and bleached under the following conditions:

	White Oak	Aspen
Cooking conditions		
Active alkali (NaOH), %	25	25
Sulfidity, %	30	30
Cooking temperature, °C.	174	174
Cooking time, min.	120	120
Yield, %	45.5	52.7
Kappa number	14.9	8.0
Bleaching conditions		
Chlorination, min.	60	60
Caustic extraction, min.	60	60
Chlorine dioxide, min.	135	150
Final pH	3.7	4.3
Yield, %	95.6	97.8
Brightness	79.3	83.3

After beating, these pulps were classified with yields as shown in Table V.

TABLE V

FIBER CLASSIFICATION DATA

	Classified Fibers Recovered, %	Fines Recovered, %	Overall Recovery, %
White oak:			
Unbeaten	80.0	12.3	92.3
10-Min. beating	84.7	5.1	89.8
20-Min. beating	76.2	10.3	86.5
Aspen:			
Unbeaten	88.5	4.0	92.5
10-Min. beating	92.0	5.8	97.8
20-Min. beating	88.5	11.5	100.0
Western softwood bleached sulfite:			
5-Min. beating	93.1	6.3	99.4
20-Min. beating	85.7	7.3	93.0
40-Min. beating	79.3	17.1	96.4

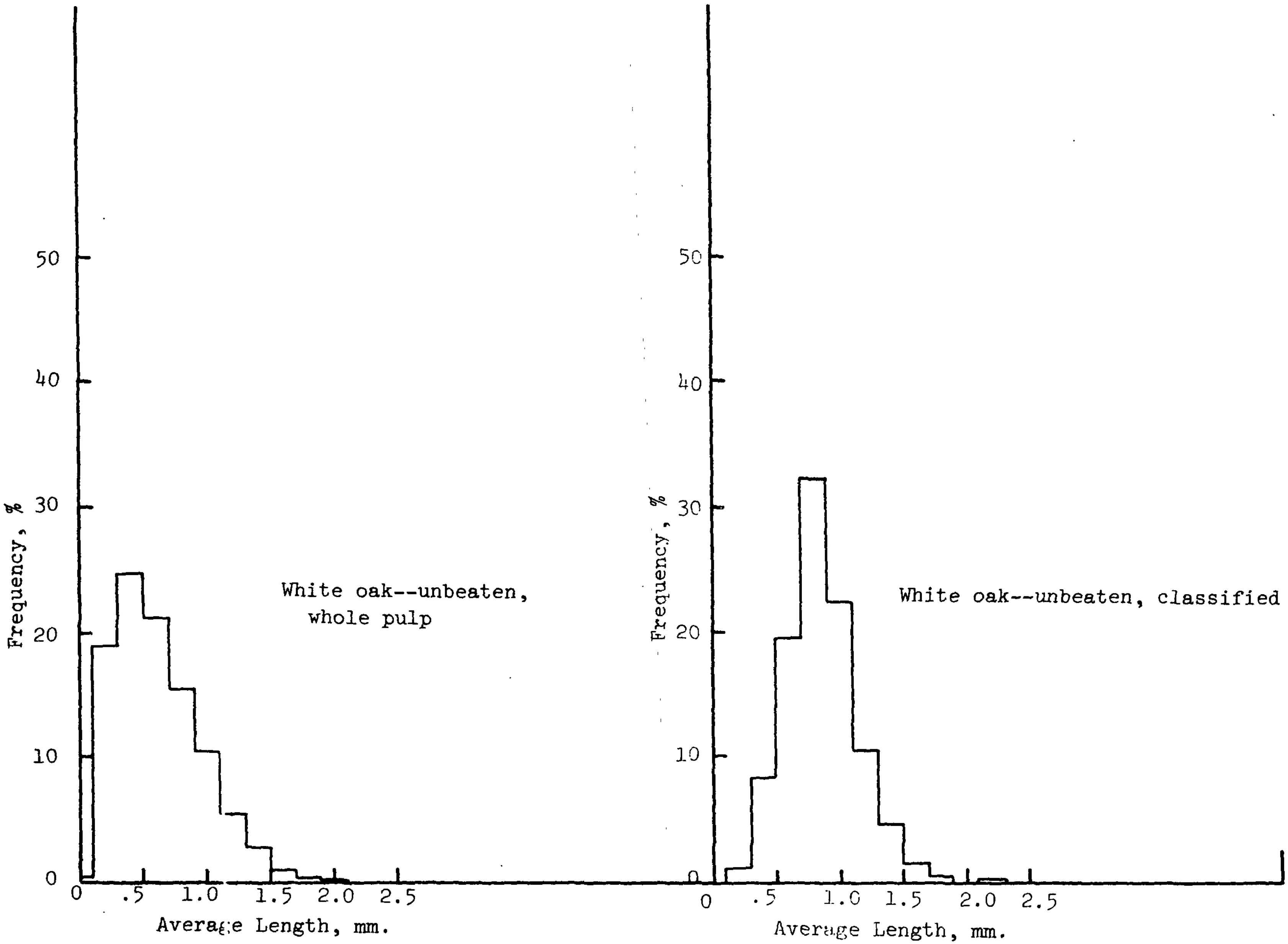

Figure 10. Fiber Length Distribution for Unbeaten White Oak

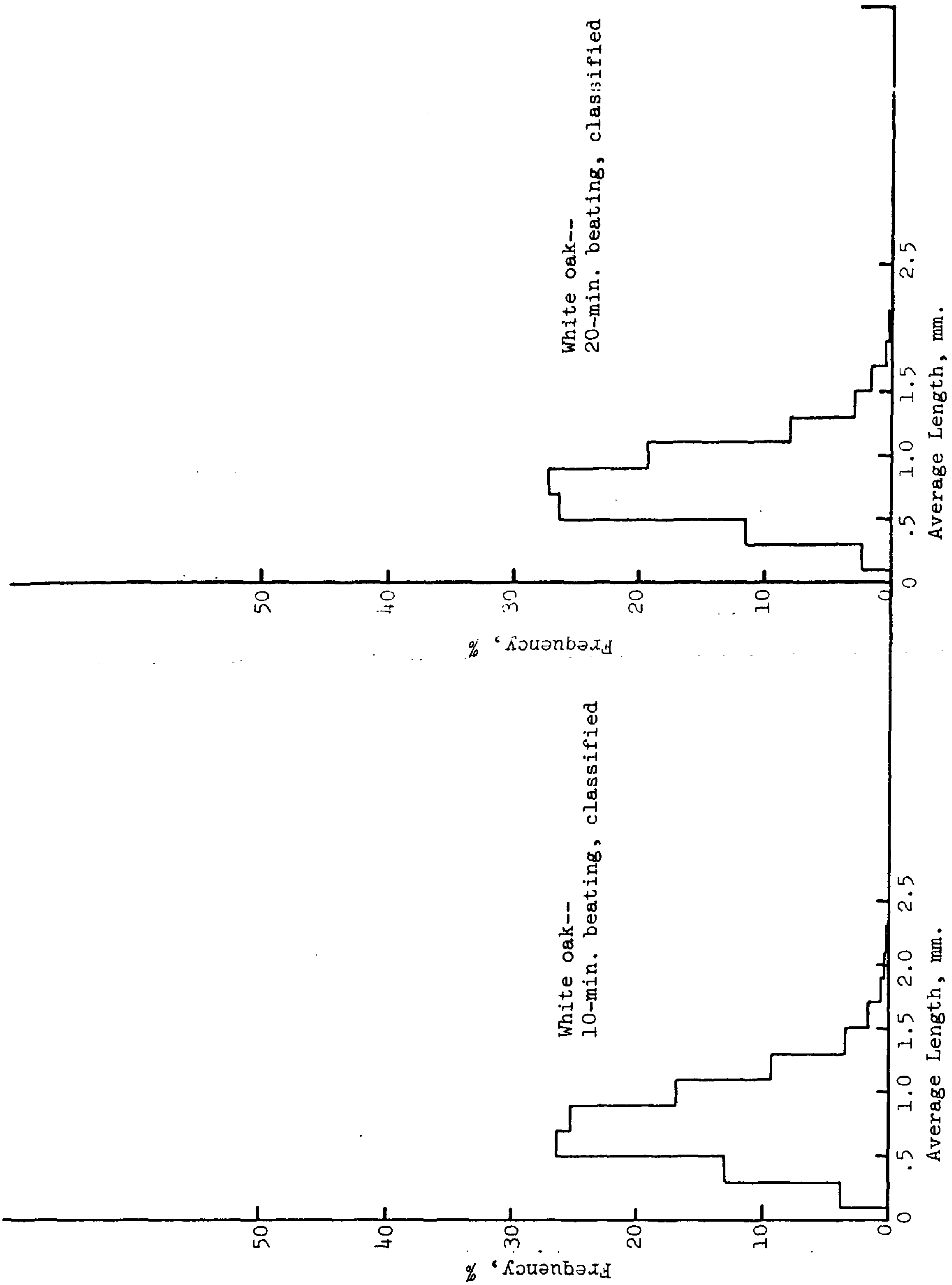

Figure 11. Fiber Length Distribution for Beaten White Oak

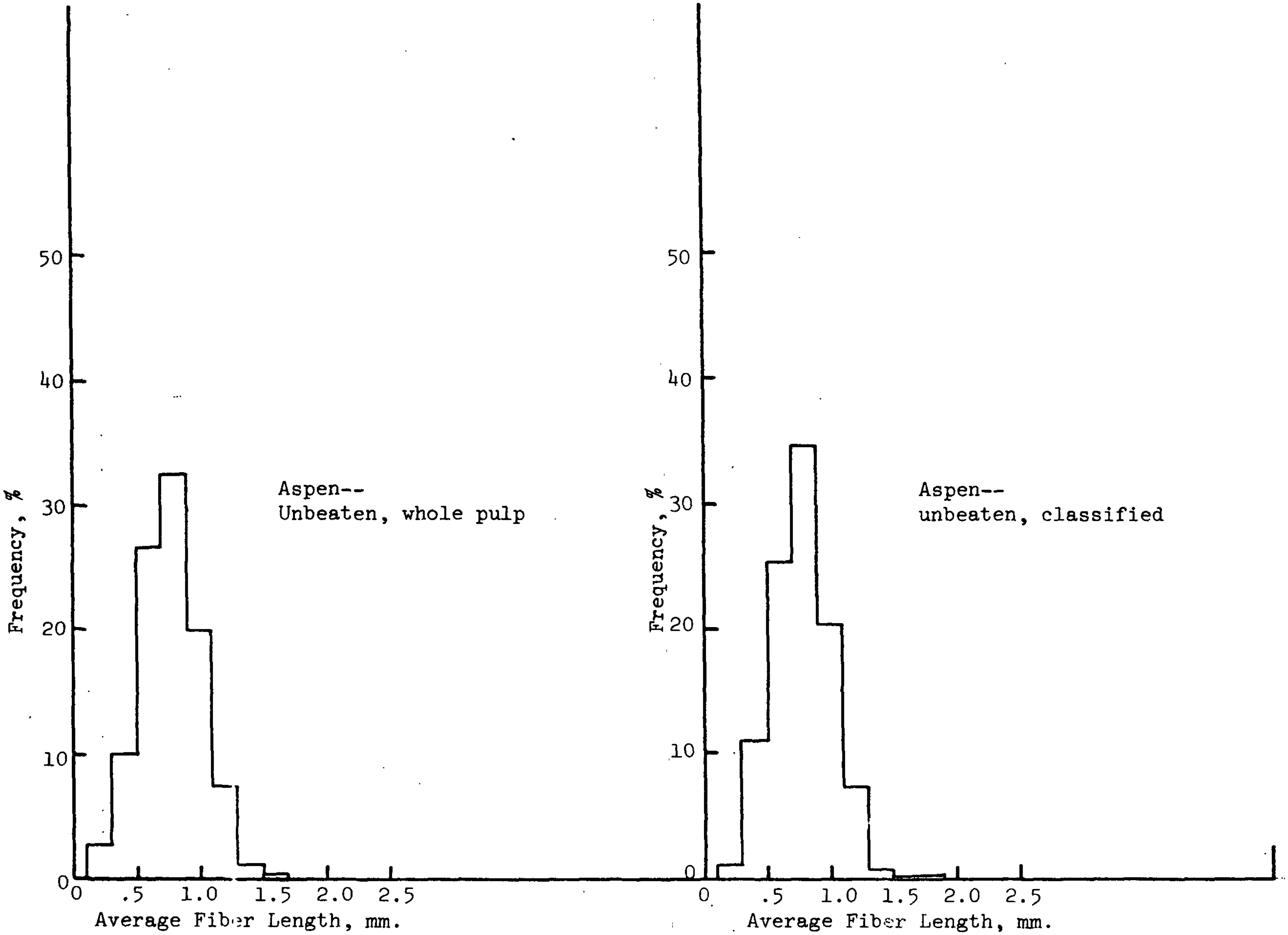

Figure 12. Fiber Length Distribution for Unbeaten Aspen

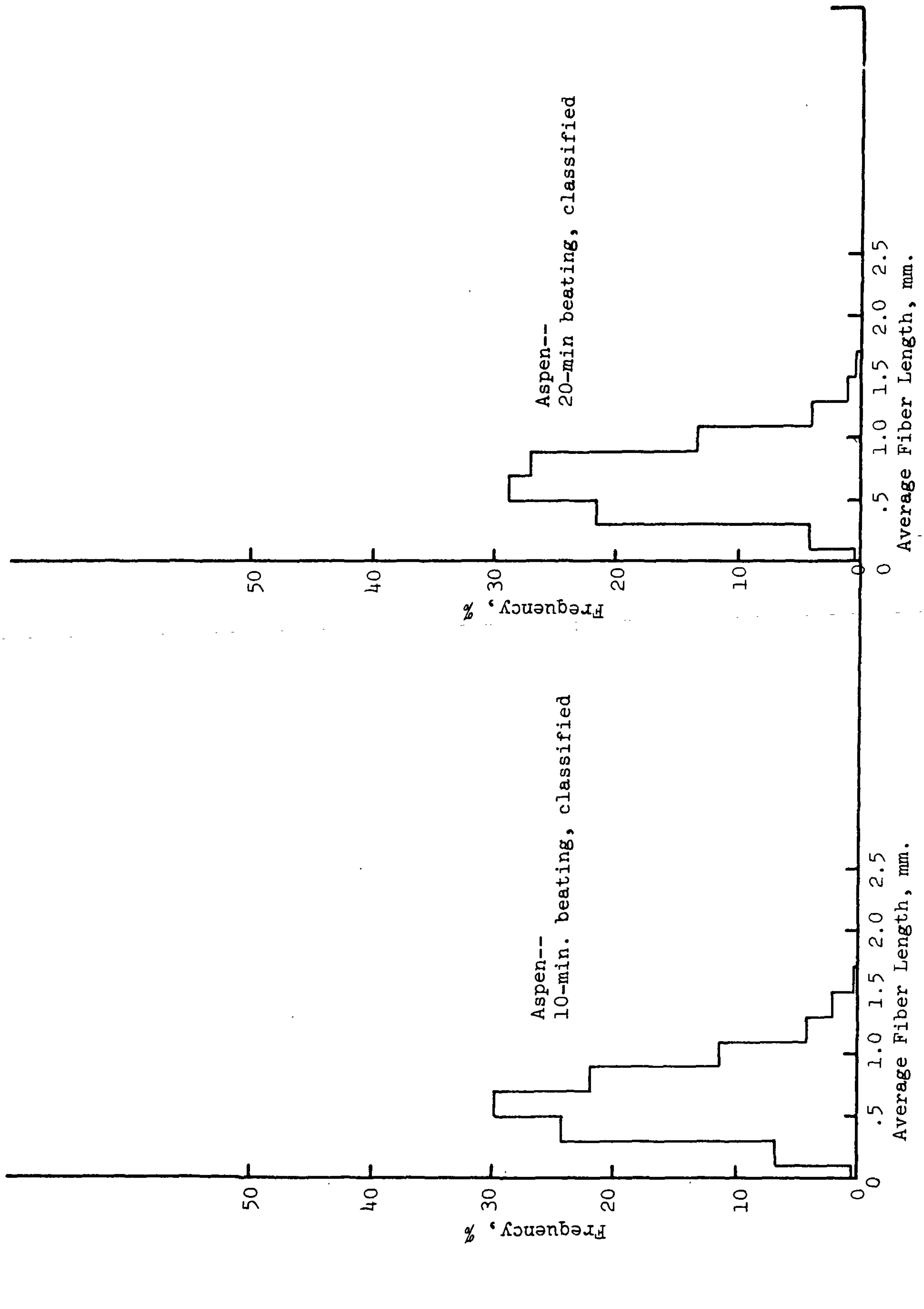

Figure 13. Fiber Length Distribution for Beaten Aspen

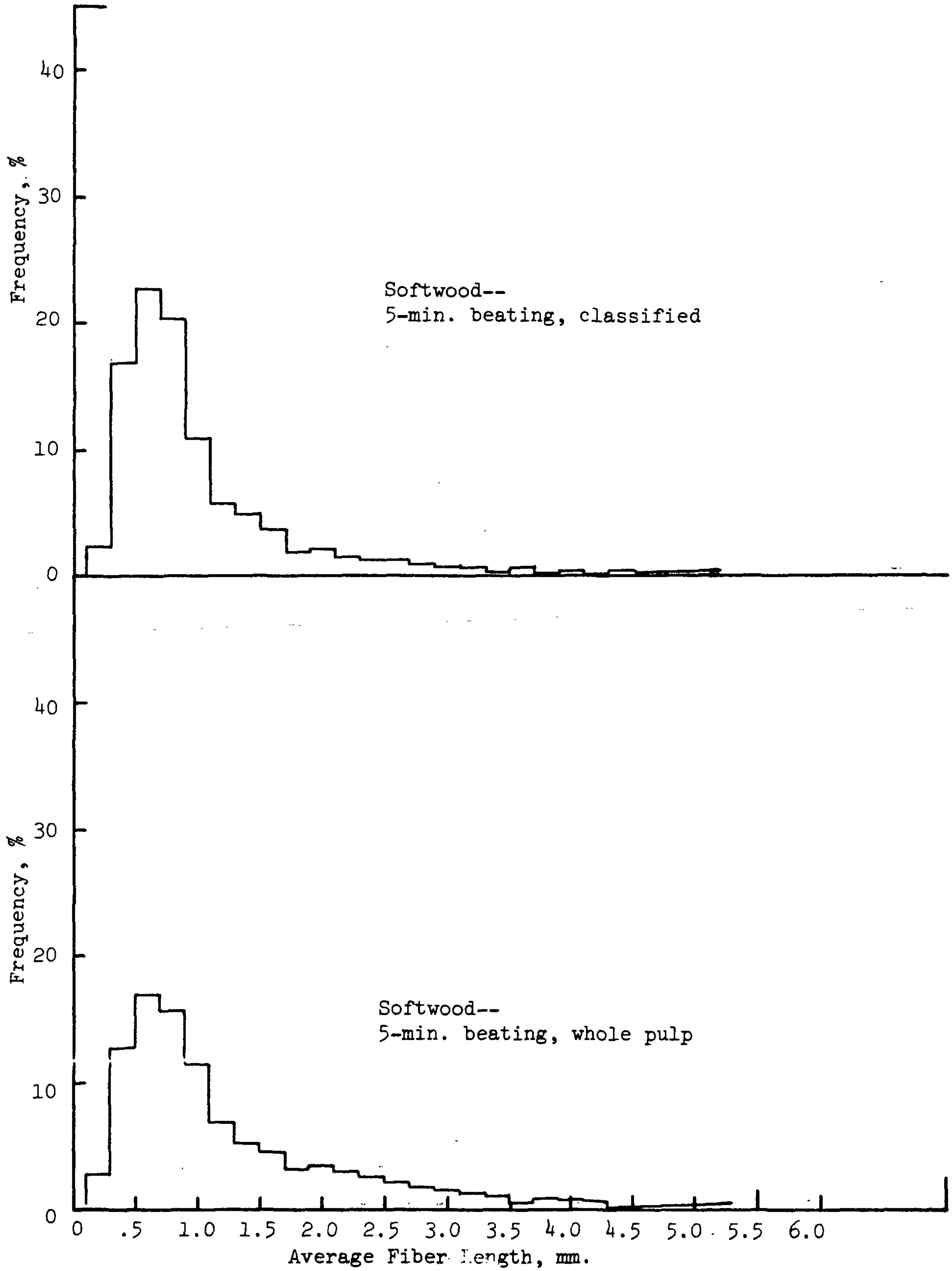

Figure 14. Fiber Length Distribution for Softwood at 5-Min. Beating

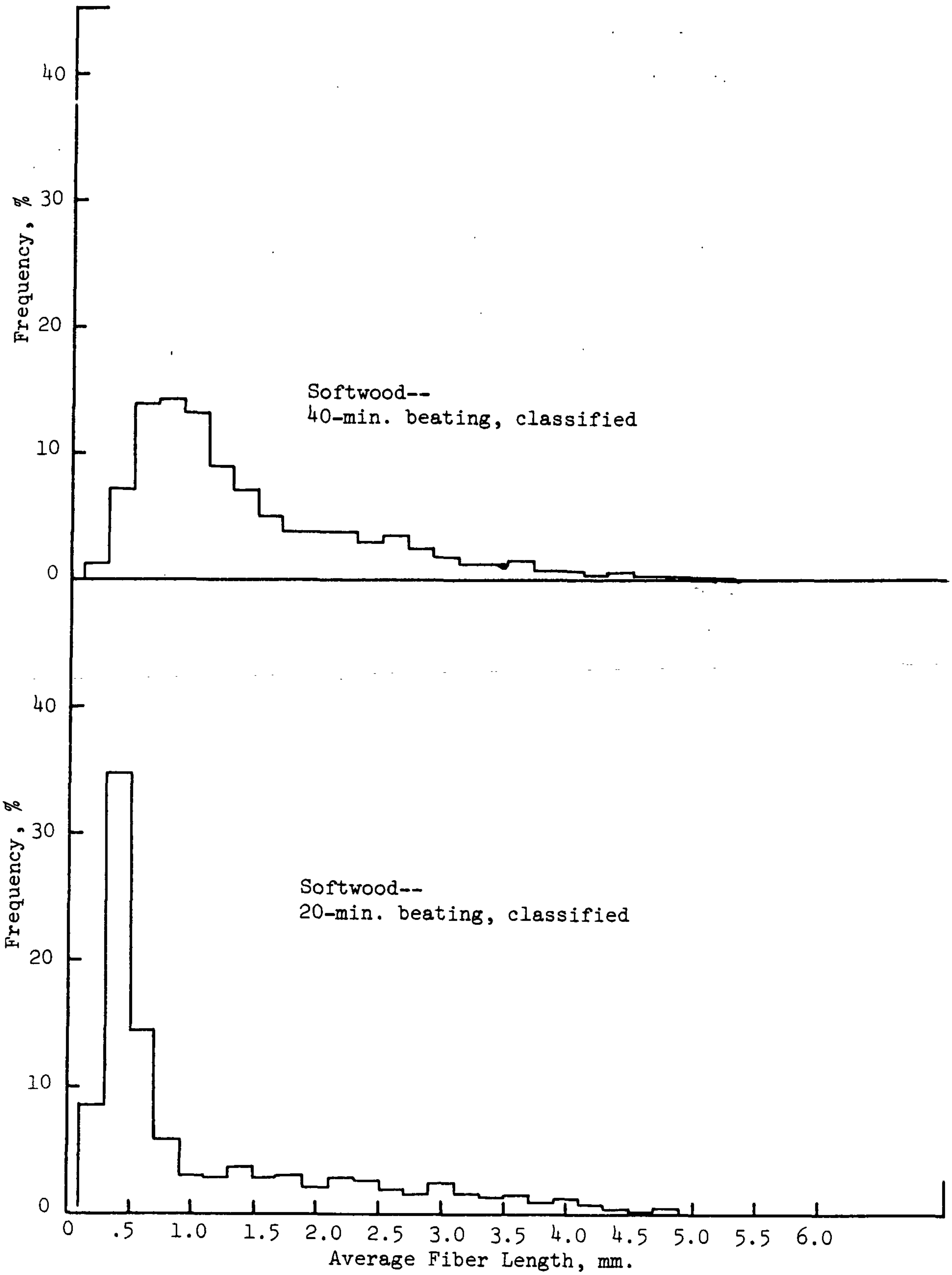

Figure 15. Fiber Length Distribution for Softwood at 20- and 40-Min. Beating

TABLE VI

ZERO-SPAN BREAKING LENGTH, METERS

Pulp	Pressing Load, p.s.i.	Beating, min. 0	Beating, min. 10	Beating, min. 20
White oak	0	11,300	12,600	13,300
	1	11,500	12,400	12,900
	5	13,300	13,000	14,200
	10	13,300	13,300	13,500
	25	14,100	13,600	13,900
	50	14,100	14,100	14,200
Aspen	0	11,900	12,000	13,600
	1	11,800	12,300	14,100
	5	13,400	13,600	15,100
	10	13,900	14,200	15,100
	25	14,900	14,800	15,600
	50	15,200	14,500	15,100

		Beating, min. 5	Beating, min. 20	Beating, min. 40
Softwood	0	11,800	13,400	14,300
	1	11,900	13,600	14,400
	5	12,400	14,500	15,100
	10	13,100	14,500	15,500
	25	13,500	14,300	14,500
	50	13,000	14,600	15,400

TABLE VII

FIBER LENGTH AND COARSENESS VALUES

Pulp	Arithmetic Average Fiber Length, mm.	Coarseness, mg./100 m.
White oak:		
Unbeaten, whole pulp	0.62	21.4
Unbeaten, classified	0.85	14.9
10-Min. beating, classified	0.79	15.6
20-Min. beating, classified	0.80	14.0
Aspen:		
Unbeaten, whole pulp	0.77	23.0
Unbeaten, classified	0.77	18.9
10-Min. beating, classified	0.65	19.8
20-Min. beating, classified	0.68	22.2
2406-A:		
5-Min. beating, whole pulp	1.14	33.4
5-Min. beating, classified	1.42	30.6
20-Min. beating, classified	1.24	29.6
40-Min. beating, classified	1.00	33.1

APPENDIX II

HANDSHEET DATA

TABLE VIII

STRENGTH PROPERTIES

Pulp	Wet Pressing, p.s.i. (nominal)	Basis Weight, g./m.²	Caliper, µm.	Tensile Strength: Breaking Length, m.	Tensile Strength: Stretch, %	Tensile Strength: Extensional Stiffness, kg./cm.²	In-Plane Tear, g.-cm./5 cm.	Elmendorf Tear, g./100 g.: One Ply	Elmendorf Tear, g./100 g.: Five Ply
White oak									
Unbeaten:	0	61.0	373	539	0.4	97	27	12	11
	1	59.6	323	719	0.4	143	37	12	11
	5	59.3	249	1140	0.6	176	46	12	17
	10	59.4	234	1260	0.7	188	54	18	24
	25	60.2	198	1660	0.9	221	72	15	25
	50	60.0	165	1930	1.0	238	113	20	32
10-Min. beating:	0	60.4	290	1470	1.2	166	107	24	31
	1	60.9	244	1750	1.5	184	124	27	36
	5	61.0	190	2310	1.7	233	153	30	46
	10	61.3	180	2380	1.7	233	160	34	53
	25	61.6	155	2730	1.9	253	181	38	58
	50	61.4	135	3040	2.1	283	201	44	66
20-Min. beating:	0	60.5	240	2400	2.2	212	177	45	54
	1	60.8	207	2610	2.3	238	205	46	62
	5	61.8	160	3430	2.8	287	258	55	71
	10	60.3	154	3480	2.9	289	249	53	75
	25	61.1	131	3820	3.0	311	286	61	85
	50	61.4	113	4270	3.4	339	306	67	93
Aspen									
Unbeaten:	0	59.5	303	1310	0.7	205	56	18	25
	1	60.2	270	1570	0.7	241	73	22	25
	5	60.8	201	2320	0.9	386	95	21	34
	10	61.4	193	2360	0.9	407	95	23	31
	25	61.3	149	2520	1.0	389	130	31	41
	50	61.1	128	3720	1.2	451	152	30	46
10-Min. beating:	0	60.3	247	2250	1.0	305	99	30	26
	1	60.7	207	2580	1.1	326	129	35	71
	5	61.0	158	3440	1.2	409	152	36	46
	10	60.7	140	3780	1.3	440	161	36	53
	25	61.3	119	4500	1.6	507	195	41	52
	50	61.2	107	5040	1.7	556	200	46	59
20-Min. beating:	0	59.9	213	2950	1.6	308	150	40	53
	1	60.8	180	3350	1.7	365	183	41	44
	5	61.3	134	4560	1.8	500	210	44	56
	10	60.5	129	4530	1.8	463	214	46	60
	25	61.0	107	5280	1.9	527	225	49	62
	50	60.7	97	5940	2.1	485	233	48	56
2406-A									
5-Min. beating:	0	57.9	261	1230	1.3	161	190	62	93
	1	61.4	221	1400	1.2	190	246	73	111
	5	61.2	188	1690	1.4	204	250	70	131
	10	60.9	172	1740	1.4	204	289	76	140
	25	58.8	140	2090	1.6	235	301	87	162
	50	59.6	127	2240	1.6	259	320	92	156
20-Min. beating:	0	60.9	213	2810	2.0	282	381	100	167
	1	60.9	183	2940	2.0	292	401	103	153
	5	61.0	145	3340	2.0	329	426	111	166
	10	61.9	137	3550	2.2	327	454	108	166
	25	60.6	114	3970	2.6	363	480	112	142
	50	61.5	109	4100	2.4	388	463	106	145
40-Min. beating:	0	61.1	176	3990	2.4	311	430	110	146
	1	61.8	157	3790	2.4	310	491	113	138
	5	61.1	125	4430	2.4	373	463	115	134
	10	61.9	118	4730	2.4	378	496	108	129
	25	61.5	108	5050	2.6	428	483	101	120
	50	61.7	102	5190	2.6	406	501	100	117

TABLE IX

SPECIFIC SURFACES AND RELATIVE BONDED AREAS

Pulp Sample	Set No.	Wet Pressing Load, p.s.i.	Specific Surface, $m.^2/g.$	Relative Bonded Area, %
White oak:				
Unbeaten	-	Unbonded fibers	0.971	--
	1	0	0.854	12
	2	1	0.905	7
	3	5	0.820	16
	4	10	0.845	13
	5	25	0.813	16
	6	50	0.781	20
10-Min. beating	-	Unbonded fibers	0.960	--
	1	0	0.799	17
	2	1	0.781	19
	3	5	0.756	21
	4	10	0.731	24
	5	25	0.727	24
	6	50	0.718	25
20-Min. beating	-	Unbonded fibers	0.900	--
	1	0	0.752	16
	2	1	0.738	18
	3	5	0.726	19
	4	10	0.743	17
	5	25	0.723	20
	6	50	0.730	19
Aspen:				
Unbeaten	-	Unbonded fibers	1.262	--
	1	0	1.035	18
	2	1	1.02	19
	3	5	1.05	17
	4	10	0.973	23
	5	25	0.911	28
	6	50	0.832	34
10-Min. beating	-	Unbonded fibers	1.221	--
	1	0	0.944	23
	2	1	0.876	28
	3	5	0.867	29
	4	10	0.813	33
	5	25	0.770	37
	6	50	0.752	38
20-Min. beating	-	Unbonded fibers	1.217	--
	1	0	0.969	20
	2	1	0.879	28
	3	5	0.777	36
	4	10	0.794	35
	5	25	0.718	41
	6	50	0.622	49
2406-A:				
5-Min. beating	-	Unbonded fibers	0.786	--
	1	0	0.655	17
	2	1	0.641	18
	3	5	0.631	20
	4	10	0.654	17
	5	25	0.605	23
	6	50	0.545	31
20-Min. beating	-	Unbonded fibers	0.709	--
	1	0	0.578	18
	2	1	0.574	19
	3	5	0.546	23
	4	10	0.606	15
	5	25	0.536	24
	6	50	0.501	29
40-Min. beating	-	Unbonded fibers	0.798	--
	1	0	0.588	26
	2	1	0.527	34
	3	5	0.516	35
	4	10	0.531	33
	5	25	0.509	36
	6	50	0.501	37

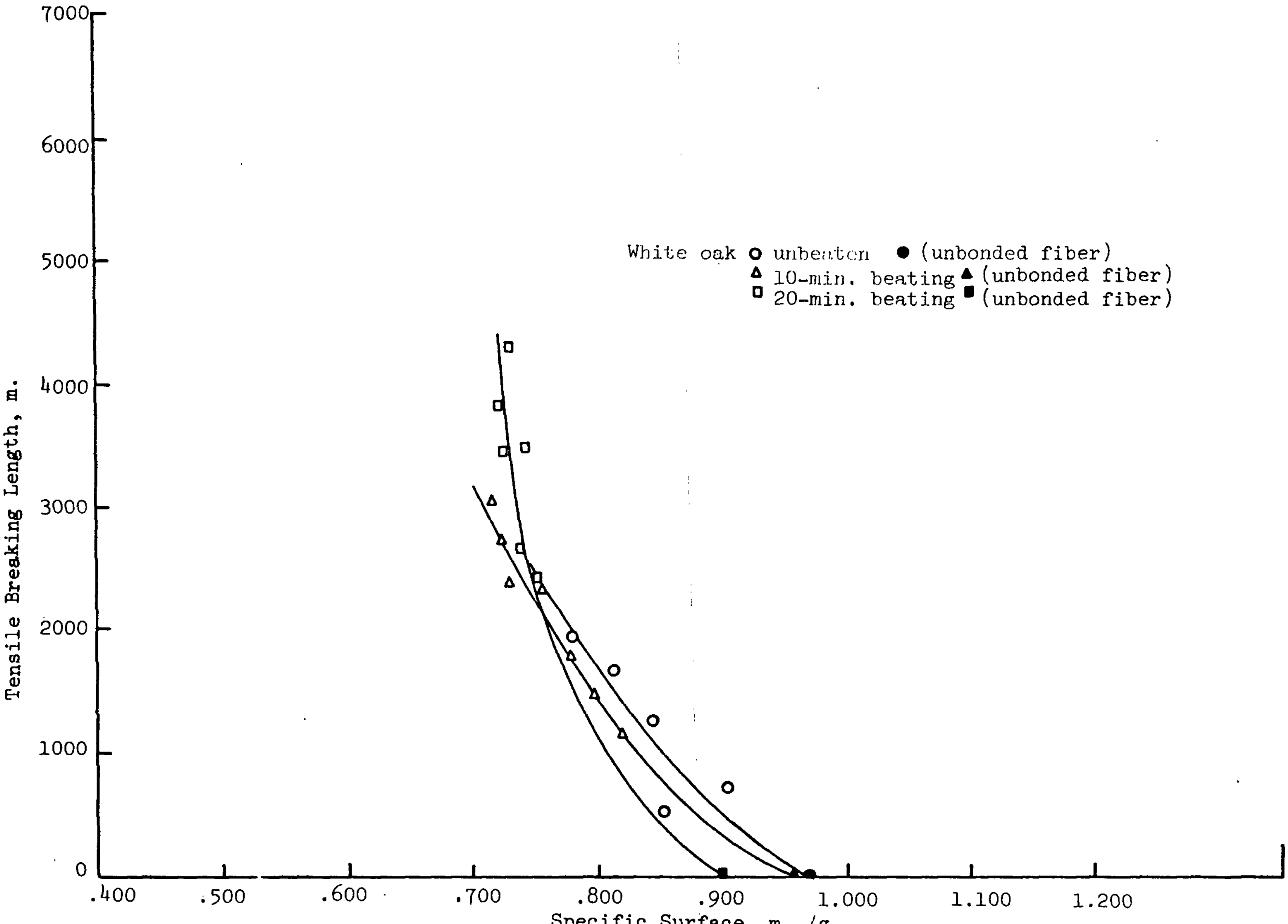

Figure 16. The Relation of Tensile Breaking Length to Specific Surface for White Oak

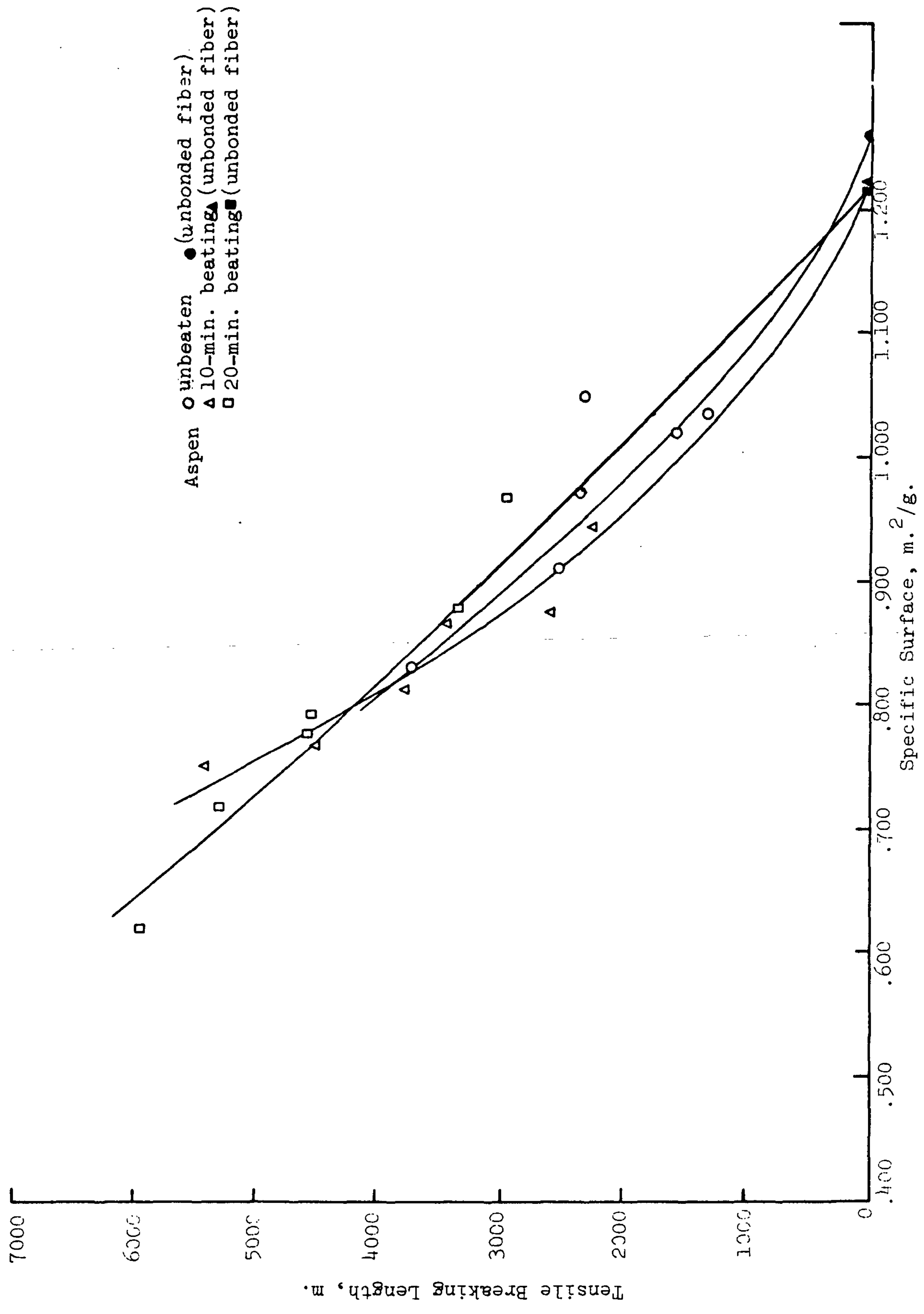

Figure 17. The Relation of Tensile Breaking Length to Specific Surface for Aspen

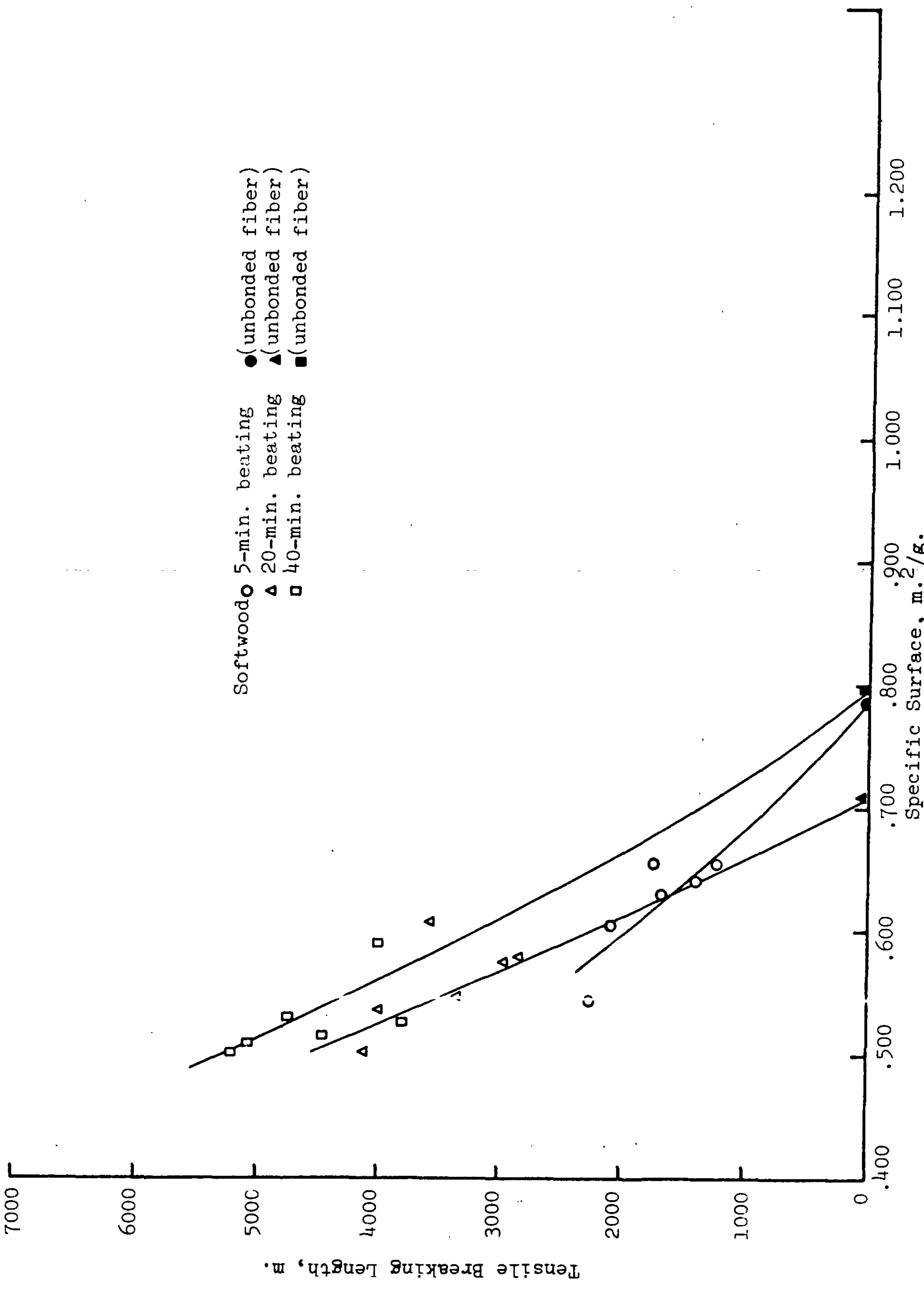

Figure 18. The Relation of Tensile Breaking Length to Specific Surface for Softwood

APPENDIX III

DYEING OF FIBERS

Previous work at the Institute dating back fifteen years (7), had indicated that fibers could be dyed under conditions that would not significantly affect the physical behavior of the fibers. Such techniques, however, should entail a minimum of treatment that might alter the fiber properties. Most fiber dyeing procedures involve heating and/or the use of salt to "set" the dye. Since these treatments might be expected to modify the fiber it was decided to forego them if possible. Some advantage with the use of fluorescent dyes had been indicated from the previous work. Special setting procedures could be obviated by thorough washing of the dyed fibers; the use of an ultraviolet light source for viewing would provide a dark background giving more contrast to the fluorescing fibers and, in the region of a tear, fibers within the sheet might be sufficiently visible that the need for a transparentizing solvent would be eliminated. Thus, the use of a fluorescent dye would facilitate the observation of the large number of specimens considered in this experimental program.

After some preliminary experimentation it was found that satisfactory results could be obtained with Calcofluor White CX Liquid applied to the fibers in the following manner:

1. Weigh out five grams of fiber (ovendry basis) and dilute to one liter with distilled water.
2. Add 2-1/2 cc. of Calcofluor White CX and agitate for 30 minutes with a laboratory mixer.
3. Filter onto No. 1 Whatman filter paper in a large Büchner funnel, redisperse and wash four times.

4. Dilute to one liter with distilled water, check consistency, and form one handsheet according to TAPPI Method T 205 for tensile strength tests. (The tensile strength of this sheet was compared to the tensile strength of a similar sheet containing 1% dyed fibers to ascertain that the dyeing procedure did not materially affect the fiber properties.)
5. Dilute 1.5 g. to 3 liters so that the addition of 25 cc. to the furnish of a sheet made by the TAPPI method will give the sheet a dyed fiber content of one percent.